



## **Deliverable 5.1: State-of-the-art on innovative non-destructive techniques, destructive techniques, scaling factors for use cases**

Work Package 5



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**EURAD-2** Deliverable 5.1 – State-of-the-art on innovative non-destructive techniques, destructive techniques, scaling factors for use cases

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## Executive Summary

This report presents a comprehensive review of the state-of-the-art of techniques used to characterise Low-Level and Intermediate-Level radioactive waste (LILW) from nuclear operations and facilities. The report catalogues and details various assessment methodologies spanning physical, chemical, radiochemical, radiological, and empirical domains. By identifying existing challenges in waste characterisation, the analysis presents solutions designed to improve measurement techniques' efficiency, accuracy, and reliability.

The report is structured around five core areas: General Waste Characterisation Approaches, Non-Destructive Techniques (NDT), Destructive Techniques (DT), Scaling Factor (SF) Methods, and Optimisation in Industrial Scenarios. The General Waste Characterisation Approaches section examines physical, chemical, and radiological characterisation methodologies, comparing NDT with DT approaches, taking into account the well-known difficult-to-measure (DTM) radionuclides and SF methods. A graded approach to characterisation is recommended, with the extent and type proportional to the potential hazard and intended management route. The NDT section covers methodologies categorised by physical, radiation-based, chemical, and radiological properties. Physical testing includes visual inspection, acoustic emission, ultrasonic testing, thermography, and liquid penetrant testing. Radiation-based methods comprise 2D/3D transmission/scatter testing, radiography, gamma inspection, neutron techniques, accelerator-based systems, muon tomography, and synchrotron characterisation. Chemical property analysis evaluates material composition. Radiological testing incorporates dose rate measurements, contamination assessment, neutron interrogation, and gamma spectrometry. Modern data management approaches include automated systems, digital twin technology, standardised formats, and blockchain storage. The review highlights challenges with heterogeneous waste matrices and the potential of Artificial Intelligence (AI)-driven data analysis. In the DT section, the report details radiochemical separation and analysis methods for DTM radionuclides, including procedures for key isotopes like  $^{14}\text{C}$ ,  $^{36}\text{Cl}$ ,  $^{41}\text{Ca}$ , and  $^{99}\text{Tc}$ . Matrix-specific applications for liquid waste, solid materials, and mixed waste are presented, along with international experience in implementing these techniques. The SF Methods section analyses the theoretical foundations and empirical applications of SF methodologies in line with ISO 21238:2007 and IAEA guidance. Statistical approaches, uncertainty quantification, and validation procedures are discussed, with emphasis on achieving regulatory compliance while avoiding excessive conservatism. The Optimisation in Industrial Scenarios section presents case studies from decommissioning projects, operational processes, and performance data demonstrating practical applications and lessons learned. Efficiency metrics, cost analysis, and safety indicators are assessed to guide optimisation strategies.

The report identifies several critical technical gaps that require further development: NDT enhancement, Physical-Chemical characterisation, DTM radionuclides analysis, and SF methodology. Regarding NDT Enhancement, limitations in detection sensitivity, processing speed, geometry handling, and data analysis automation need to be addressed for more efficient waste characterisation. For Physical-Chemical characterisation, improvements are needed in real-time capabilities, non-destructive methods, automation levels, and cost efficiency. DTM radionuclides analysis faces challenges in sample preparation time, detection limits, matrix interference, and analysis costs that continue to constrain the execution of a comprehensive waste characterisation. SF methodology would benefit from refinements in statistical accuracy, validation procedures, correlation reliability, and uncertainty quantification to enhance SF applications.

## Keywords

Radioactive Waste Characterisation, Low-Level Waste, Intermediate-Level Waste, Decommissioning, Non-Destructive Techniques, Destructive Techniques, Scaling Factor, Difficult-to-Measure Radionuclides, Gamma Spectrometry, Neutron Interrogation, Radiochemical Separation, Data Management Automation, Uncertainty Quantification, Waste Acceptance Criteria.

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## Glossary

**Accelerator Mass Spectrometry (AMS):** A highly sensitive analytical technique that separates and quantifies isotopes by accelerating ions to high energies and measuring their mass-to-charge ratios.

**Alpha Spectrometry:** Analytical technique used to identify and measure the concentrations of alpha-emitting radionuclides by detecting the energy of emitted alpha particles.

**Beta Emitter:** A radionuclide that decays by emission of beta particles (electrons or positrons).

**Destructive Techniques (DT):** Characterisation methods that require sampling and modification or destruction of the sample during analysis, typically involving radiochemical separations.

**Difficult-to-Measure (DTM) Radionuclides:** Nuclides whose radioactivity cannot be directly assessed using non-destructive methods, such as alpha or beta emitters that require radiochemical separation before measurement.

**FEFF:** Widely used ab initio code for calculating X-ray absorption spectroscopy, X-ray absorption near-edge structure, extended X-ray absorption fine structure and various other spectra for clusters of atoms.

**FDMNES (Finite Difference Method Near Edge Structure):** Simulation code specifically designed for calculating X-ray absorption and emission spectra, particularly in the near-edge region.

**Gamma Spectrometry:** Analytical technique that identifies radionuclides by measuring the energy and intensity of gamma rays emitted during radioactive decay.

**High Level Waste (HLW):** Waste which contains such large concentrations of both short-lived and long-lived radionuclides that, compared to intermediate level waste, requires a greater degree of containment and isolation from the accessible environment. Such isolation is likely to require engineered barriers and natural barriers in a stable deep geological formation

**Heterogeneous Waste:** Waste that varies in composition and/or activity distribution throughout its volume, presenting challenges for representative sampling and accurate characterisation.

**Integral Gamma Scanning (IGS):** Non-destructive measurement technique that uses an open or collimated detection geometry to acquire an integrated gamma spectrum of a waste package.

**Key Nuclides:** Gamma-emitting radionuclides that can be measured easily using non-destructive methods (e.g.,  $^{60}\text{Co}$  and  $^{137}\text{Cs}$ ) and exhibit correlations with difficult-to-measure nuclides.

**Key radionuclides:** see Key Nuclides.

**Legacy Waste:** Historical radioactive waste with limited documentation on composition and characteristics, often presenting significant characterisation challenges.

**Liquid Scintillation Counting (LSC):** Analytical technique used to measure the activity of alpha and beta-emitting radionuclides by detecting light pulses produced when radiation interacts with a scintillation cocktail.

**Low level waste (LLW):** Waste that is above clearance levels, but with limited amounts of long lived radionuclides. Such waste requires robust isolation and containment for periods of up to a few hundred years and is suitable for disposal in engineered near surface facilities. This class covers a very broad range of waste. LLW may include short lived radionuclides at higher levels of activity concentration, and also long lived radionuclides, but only at relatively low levels of activity concentration.

**Intermediate level waste (ILW):** Waste that, because of its content, particularly of long lived radionuclides, requires a greater degree of containment and isolation than that provided by near surface disposal. However, ILW needs no provision, or only limited provision, for heat dissipation during its storage and disposal. ILW may contain long lived radionuclides, in particular, alpha emitting radionuclides that will not decay to a level of activity concentration acceptable for near surface disposal during the time for which institutional controls can be relied upon.

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**Matrix Effects:** Influence of the waste material composition (matrix) on measurement results, particularly attenuation of radiation in dense or heterogeneous materials.

**Minimum Detectable Activity (MDA):** The lowest activity that can be detected with a specified degree of confidence using a particular measurement system and technique.

**Non-Destructive Techniques (NDT):** Characterisation methods that do not require sampling or alteration of the waste package, typically based on measuring radiation emissions from intact containers.

**Radiochemical Separation:** Chemical processes used to isolate specific radionuclides from sample matrix and interfering nuclides prior to measurement, essential for analyzing difficult-to-measure nuclides.

**Scaling Factor (SF):** Mathematical parameter used to calculate the activity of difficult-to-measure radionuclides based on measured activities of key nuclides, utilising established correlations.

**Segmented Gamma Scanning (SGS):** Non-destructive technique that measures gamma emissions from discrete segments of a waste package using collimated detectors to create vertical activity profiles.

**Tomographic Gamma Scanning (TGS):** Advanced form of gamma scanning that creates three-dimensional maps of activity distribution within waste packages by combining transmission and emission measurements.

**Transuranic Elements (TRU):** Elements with atomic numbers greater than uranium (92), including plutonium, americium, and curium, typically alpha emitters with long half-lives.

**Validation:** Process of confirming that analytical methods or SF provide results that meet specified requirements for accuracy and reliability.

**Waste Acceptance Criteria (WAC):** Set of requirements that radioactive waste packages must meet for acceptance at storage or disposal facilities, defining limits on physical, chemical, and radiological properties.

**Waste Package:** Container with its radioactive contents prepared for handling, transport, storage and/or disposal; may be a metal drum, concrete container, or other engineered containment system.

**Work Package (WP):** A defined component of a project with specific deliverables, activities, and resources.

## Key Abbreviations

AAS	-	Atomic Absorption Spectroscopy
AE	-	Acoustic Emission
AI	-	Artificial Intelligence
AMS	-	Accelerator Mass Spectrometry
AMP-PAN	-	Ammonium molybdophosphate-polyacrylonitrile
ANI	-	Active Neutron Interrogation
ANNs	-	Artificial Neural Networks
ASR	-	Alkali-Silica Reaction
BIM	-	Building Information Modeling
BWR	-	Boiling Water Reactor
CLEANDEM	-	Cyber physical Equipment for unManned Nuclear DEcommissioning Measurements
CMT	-	Cemented Waste
CRDS	-	Cavity Ring-Down Spectroscopy
DDA	-	Differential Die-Away Analysis
DMG	-	Dimethyl glyoxime
DTW	-	Digital Twin
DT	-	Destructive Techniques
DTM	-	Difficult-To-Measure (radionuclides)
EC	-	Electron Capture
EOSC	-	European Open Science Cloud
ETM	-	Easy-To-Measure (radionuclides)
EURAD	-	European Joint Programme on Radioactive Waste Management
EURAD-2	-	European Partnership on Radioactive Waste Management-2
FAIR	-	Findable, Accessible, Interoperable, and Reusable
FDMNES	-	Finite Difference Method Near Edge Structure
GM	-	Geiger–Müller
GPR	-	Ground Penetrating Radar
GPS	-	Global Positioning System
HLW	-	High Level Waste
HPGe	-	High-Purity Germanium (detector)
HTTPS	-	Hypertext Transfer Protocol Secure
IAEA	-	International Atomic Energy Agency
ICP-MS	-	Inductively Coupled Plasma Mass Spectrometry

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IGS	-	Integral Gamma Scanning
ILW	-	Intermediate-Level Waste
INSIDER	-	Improved Nuclear Site characterisation for waste minimisation in Decommissioning and Dismantling operations under constrained Environment
ISO	-	International Organisation for Standardisation
KN	-	Key Nuclide
LIBS	-	Laser-Induced Breakdown Spectroscopy
LILW	-	Low and Intermediate Level Waste
LLW	-	Low-Level Waste
LoRaWAN	-	Long Range Wide Area Network
LSC	-	Liquid Scintillation Counting
MDA	-	Minimum Detectable Activity
MICADO	-	Measurement and Instrumentation for Cleaning and Decommissioning Operations
ML	-	Machine Learning
MQTTS	-	Message Queuing Telemetry Transport Secure
NAA	-	Neutron activation analysis
NDT	-	Non-Destructive Techniques
NEA	-	Nuclear Energy Agency
NEXAFS	-	Near-edge X-ray Absorption Fine Structure
NRG	-	Nuclear Research and Consultancy Group
NPP	-	Nuclear Power Plant
OECD	-	Organisation for Economic Co-operation and Development
PLEIADES	-	Platform based on Emerging and Interoperable Applications for enhanced Decommissioning processes
PREDIS	-	Pre-Disposal Management of Radioactive Waste
PROV-0	-	Provenance Ontology
PWR	-	Pressurised Water Reactor
QC	-	Quality Control
RW	-	Radioactive Waste
RBMK	-	Reaktor Bolshoy Moshchnosti Kanalnyy (High Power Channel-type Reactor)
RepMet	-	Radioactive Waste Repository Metadata Management
RIMS	-	Ionisation Mass Spectrometry
RT	-	Radiation-based Testing
RWM	-	Radioactive Waste Management
RWP	-	Radioactive Waste Package
SF	-	Scaling Factor

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SGS	-	Segmented Gamma Scanning
SSN	-	Semantic Sensor Network
STXM	-	Scanning Transmission X-ray Microscopy
TBP	-	Tributyl phosphate
TGS	-	Tomographic Gamma Scanning
TIMS	-	Thermal Ionisation Mass Spectrometry
TRL	-	Technology Readiness Level
TRU	-	Transuranic Elements
UT	-	Ultrasonic Testing
UGV	-	Unmanned Ground Vehicle
WAC	-	Waste Acceptance Criteria
WP	-	Work Package
WP5 ICARUS	-	Work Package 5: ICARUS (Innovative Characterisation Techniques for Large Volumes)
XAS	-	X-ray Absorption Spectroscopy
XAFS	-	X-ray Absorption Fine Structure
XANES	-	X-ray Absorption Near-Edge Structure
XRD	-	X-Ray Diffraction
XRF	-	X-Ray Fluorescence
QQQ-ICP-MS	-	Triple quadrupole inductively coupled plasma mass spectrometry

## 1. Introduction

The European Partnership on Radioactive Waste Management-2 (EURAD-2) Work Package 5 (WP5) – ICARUS (Innovative Characterisation Techniques for Large Volumes) focuses on advancing, optimising and harmonising cutting-edge techniques for characterising the radiological, physical, and chemical properties of low and intermediate-level mixed radioactive waste (LILW). These characterisation capabilities are essential for ensuring safe implementation of radioactive waste management programs across Europe. The research integrates laboratory-scale destructive techniques (DT) with field-deployable non-destructive techniques (NDT), establishing reliable correlations through scaling factors (SF) for both raw waste materials and packaged waste packages.

The objectives established for this project are important and represent a logical continuation of work previously conducted in EURAD-1 [1]. Earlier, within the PREDIS project [2], the issue of radioactive waste management prior to disposal was considered comprehensively. In particular, it was noted that radioactive waste is generated not only during the operation and decommissioning of nuclear facilities, but also through the use of radionuclides in scientific research. However, significant volumes of radioactive waste are generated specifically during electricity production by nuclear power plants.

The EURAD Roadmap identifies waste characterisation as a critical component under Theme 2: Pre-disposal, specifically in Sub-theme 2.2: "Implementing predisposal management of radioactive waste to support key risk and hazard reduction, and to help reduce costs and save space at interim storage and disposal facilities." Within this implementation framework, Section 2.2.1 focuses on the need to "Sort, characterise, classify and quantify radioactive waste in accordance with requirements established or approved by the regulatory body." [1]

According to [2], around 3.0 million m<sup>3</sup> of LILW has been generated in Europe, of which about 20% has been stored and 80% has been disposed of [3]. A significant amount of LILW, sometimes mixed, is also expected to be generated during the decommissioning of nuclear power plants. Therefore, there is a logical need to optimise the characterisation process for such large volumes of radioactive waste. An important aspect is the presence of historical radioactive waste in some European countries [6]. Information about the characteristics of such waste is typically very limited, and detailed characterisation of this waste, due to its volume and nuclide composition, may be inefficient in the context of planning further activities with this waste. Additionally, the question of radioactive waste classification arises, since different Member States use different approaches to classify and define LLW and ILW.

Within WP5 ICARUS, LILW-mixed waste will primarily refer to non-toxic waste such as glass, plastic, parts of clothes and rags, metal parts, etc., in solid phase, classified as LLW and ILW, generally packaged or conditioned, the radiological characterisation, when available, is considered poorly reliable and chemical characterisation is not available [4] (this definition differs from the IAEA's standard definition of mixed waste [5]). The radionuclides of interest typically encompass both easy-to-measure (ETM) gamma emitters (key nuclides) such as <sup>137</sup>Cs and <sup>60</sup>Co, as well as difficult-to-measure (DTM) radionuclides including alpha and pure beta emitters (e.g., <sup>14</sup>C, <sup>36</sup>Cl, <sup>41</sup>Ca, <sup>99</sup>Tc) that require destructive analysis techniques.

Which radionuclides to account for depends heavily on the waste origin (operational vs. decommissioning), facility type (research, medical, power generation), and waste management phase (processing, storage, disposal, or potential clearance). The expected activity range varies significantly, from near-clearance levels (approximately 0.1-1 Bq/g) for potentially releasable materials to several kBq/g for ILW requiring remote handling.

It is also crucial to account for uncertainties that emerge throughout the characterisation process, starting from the analysis of available information, sampling and preparation of samples for analysis, through to performing measurements using both destructive and non-destructive methods and assessment of the measurements. In this context, it is important to establish relationships between DT on laboratory scale and NDT, particularly SF, to reduce characterisation uncertainty or establish an acceptable level of conservatism for consideration in subsequent radioactive waste management. In this



context, an important issue that will be addressed within WP5 ICARUS is sampling design for accuracy improvement. As noted in [6] one general difference between sampling of legacy and non-legacy waste that should be highlighted is that for sampling of legacy wastes more protective measures need to be taken, and if the segregated legacy waste is heterogeneous, more samples need to be taken to allow a representative characterisation of the waste. The organisation of sampling of radioactive waste material for characterisation is closely linked to the representativeness of the results of this characterisation for the given radioactive waste material and the determination of SF.

Due to years of EU Member States' experience in NPP operation including predisposal waste management, there are numerous mature technologies and services available on the international market. Some countries and companies have been operating predisposal waste management facilities for decades, including interim storage and final disposal or even free release of wastes reused by other industries. Companies that are offering predisposal waste management services can be found by international trade registries, associations such as SNETP and World Nuclear Association, and via their participation at trade fairs on decommissioning and waste management. Within the market offering, it is acknowledged that there are some problematic waste streams, such as graphite materials from reactor decommissioning, which are still at the research and development stage for predisposal processing prior to disposal. The sorting, characterisation, processing and packaging of some of these types of waste is not market ready.

The Regulatory Framework governing radioactive waste characterisation and management is structured around international standards and European directives, ensuring a harmonised approach to safety, environmental protection, and long-term disposal strategies. Effective waste characterisation requires careful consideration of several key parameters that define the scope and limitations of the process. Regulatory requirements from national authorities and international bodies such as IAEA Safety Standards and EC Directives establish mandatory frameworks for comprehensive waste characterisation, while corporate requirements often include more stringent internal protocols aligned with optimisation goals and waste acceptance criteria at disposal facilities.

The guidelines are primarily developed by the International Atomic Energy Agency (IAEA) and the Nuclear Energy Agency (NEA). These organisations set forth standards that guide the safe handling, characterisation, and disposal of radioactive waste. IAEA Safety Standards establish fundamental principles for radiation protection, waste classification, storage, and disposal. Key references include IAEA-TECDOC-1537 "Strategy and Methodology for Radioactive Waste Characterisation" [8], and the IAEA General Safety Requirements (GSR Part 5) "Predisposal Management of Radioactive Waste" [7] NEA Guidelines [9] supplement international efforts by promoting best practices, research findings, and regulatory collaboration. Reports such as [9] focus on unconventional and legacy waste characterisation. These frameworks emphasise the traceability, documentation, and compliance of radioactive waste throughout its lifecycle.

At the European level, the EC Directive 2011/70/EURATOM [10] establishes a community framework for responsible and safe management of spent fuel and radioactive waste. This directive mandates that each EU member state develop national programs for waste management, including characterisation, storage, and disposal. Independent regulatory bodies ensure compliance with technical and safety standards. Transparency and public engagement are also emphasised, requiring clear communication of waste management strategies to stakeholders.

The characterisation of radioactive waste is critically important at all stages of its lifecycle. It provides the foundation for planning further waste management, allowing for resource optimisation and cost minimisation. This issue is particularly relevant for large volumes of waste generated in the nuclear energy sector. Using various characterisation methods, automating processes, and implementing innovative approaches can significantly enhance the efficiency of this process.

One direction for future work is the development, optimisation, and harmonisation of innovative methods for characterising the physical, chemical, and radiological properties of waste, including gamma activity

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analysis in large complex containers. An important tool is the implementation of SF method, as well as automated data processing systems to improve accuracy and accelerate the process.

The SF method has emerged as a primary approach for characterising radioactive waste, particularly for DTM radionuclides. According to IAEA documentation [8], this method establishes correlations between easily measurable radionuclides (key nuclides) and DTM radionuclides, allowing for indirect determination of DTM activities. The International Organisation for Standardisation has recognised the importance of this approach through ISO 21238:2007 [13], which standardises the SF methodology for low and intermediate-level radioactive waste packages generated at nuclear power plants. However, SF method implementation faces challenges relating to statistical reliability, particularly when dealing with heterogeneous waste streams or when correlation data is limited.

Modern characterisation methods often face limitations related to accuracy, data processing speed, and the need for highly sensitive technologies to determine radionuclide composition. Time and resource limitations represent significant practical challenges, as comprehensive characterisation of large waste volumes using destructive methods is prohibitively expensive and time-consuming, driving the need for efficient non-destructive techniques coupled with scaling factor methodologies. Practical constraints include the heterogeneity of waste forms affecting representative sampling, limited accessibility for measurement, radiation exposure concerns for personnel, contamination control during sampling, and the need for specialised facilities and equipment for handling higher-activity samples. Technologies used for characterisation sometimes have insufficient resolution, complicating the analysis of complex waste [11]. These parameters collectively determine the optimal characterisation approach for specific waste streams.

Despite the advanced level of characterisation methods, there are problems with implementing cutting-edge technologies in practice. First, there is a lack of unified standards for different types of waste and limited access to data necessary for method calibration. Second, integrating automated data processing systems and AI analysis requires substantial investment and technical adaptation at nuclear facilities. Technological and software solutions must meet safety and reliability requirements when operating in conditions of increased radiation hazard [12].

Segmented gamma scanning (SGS) represents another cornerstone technology for waste characterisation, allowing for non-destructive assessment of gamma-emitting radionuclides. Advanced implementations, such as tomographic gamma scanning (TGS), provide three-dimensional activity distribution information within waste packages.

The development of these areas will improve approaches to radioactive waste characterisation, make them more consistent with international standards and national regulatory requirements, and contribute to more efficient and safer radioactive waste management. This report presents a comprehensive review of the state-of-the-art characterisation techniques for large volume mixed LILW from nuclear operations and facilities. The analysis addresses current challenges in waste characterisation and proposes innovative solutions to enhance efficiency, accuracy, and reliability of measurement techniques.

## 2. General Waste Characterisation Approaches

Radioactive waste characterisation provides essential information about waste properties to ensure safe and efficient management throughout its lifecycle. According to the IAEA-TECDOC-1537 [8] effective characterisation is critical for determining appropriate treatment methods, ensuring regulatory compliance, and supporting long-term safety assessments. This chapter presents an overview of key characterisation approaches covering physical, chemical, and radiological aspects.

### 2.1 Physical Characterisation

Physical characterisation determines the material properties of radioactive waste that influence its handling, processing, and long-term behaviour. Key parameters include:

- **Density and specific gravity:** Essential for volume calculations and treatment planning
- **Particle size distribution:** Affects processing options and waste form stability
- **Porosity and permeability:** Impact potential leaching behaviour and waste form stability
- **Thermal properties:** Critical for heat-generating waste or thermal treatment processes for high level waste (HLW)
- **Mechanical strength:** Important for waste package integrity assessment

Common characterisation techniques include direct measurements (mass, volume, density), visual inspection, and advanced imaging methods such as X-ray radiography and computed tomography. These non-destructive imaging techniques can identify internal structures, voids, and heterogeneities within waste packages [14].

Physical characterisation data directly guides decisions on treatment methods, packaging designs, storage requirements, and transportation needs. IAEA Technical Reports Series No. 383 emphasises that physical properties serve as quality indicators for waste packages and provide essential input for safety assessments [15].

### 2.2 Chemical Characterisation

Chemical characterisation identifies the elemental and molecular composition of waste, including both radioactive and non-radioactive constituents. This information is crucial for:

- Evaluating potential chemical hazards
- Selecting appropriate treatment technologies
- Assessing long-term waste form behaviour
- Ensuring compatibility with disposal environments

Key chemical parameters include:

- **Elemental composition:** Major and trace elements including potentially hazardous components
- **Molecular composition:** Chemical compounds and their structures
- **pH and redox potential:** Affecting chemical stability and radionuclide speciation
- **Organic content:** Influencing waste form stability and gas generation potential
- **Corrosion potential:** Critical for metallic waste or container assessment
- **Gas generation:** assessment of chemical reactions that may lead to gas formation (e.g. hydrogen, methane, ammonia)

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- **Leaching:** determining the rate at which radionuclides may migrate from waste into the environment when in contact with water
- **Explosion safety and fire resistance:** chemical content of reactive components that could cause thermal or explosive reactions.

Analytical techniques commonly employed include spectrometric methods (X-Ray Fluorescence (XRF), Inductively Coupled Plasma Mass Spectrometry (ICP-MS), Inductively Coupled Plasma – Optical Emission Spectrometry (ICP-OES), Atomic Absorption Spectroscopy (AAS), chromatographic techniques (High-Performance Liquid Chromatography (HPLC), Liquid Chromatography-Mass Spectrometry (LC-MS), Gas Chromatography-Mass Spectrometry (GC-MS), Ion Chromatography (IC) X-ray diffraction, thermal analysis, CHNOS Elemental Analysers, etc. For characterising the specific mixed waste streams, analytical techniques must be adapted to address the challenges presented by physically heterogeneous materials while maintaining appropriate detection limits for the radionuclides of interest and accounting for potential matrix interference effects from the diverse material compositions.

A review of characterisation techniques at CEA, France highlights that no single technique provides complete chemical characterisation, necessitating complementary approaches tailored to specific waste types [16]. Chemical characterisation directly supports waste classification decisions, treatment technology selection, and performance assessment for disposal facilities. "The methodologies applied in waste characterisation and process control should guarantee the stability and integrity of waste packages. Otherwise, the long-term safety assessment of the intended disposal facility may be compromised." [8]

## 2.3 Radiological Characterisation

Radiological characterisation forms the core of radioactive waste assessment, determining the radionuclide inventory, activity concentrations, and radiation fields. This information is fundamental for:

- Waste classification according to regulatory frameworks
- Handling and shielding requirements
- Treatment and conditioning decisions
- Transport planning
- Disposal facility safety assessment

Key parameters include:

- **Radionuclide identification:** Determining specific radionuclides present
- **Activity concentration:** Quantifying activity per unit mass or volume
- **Activity distribution:** Assessing spatial distribution in heterogeneous waste
- **Dose rate measurements:** Determining external radiation fields
- **Surface contamination:** Measuring removable and fixed contamination levels

Common measurement techniques include:

- **Geiger-Mueller (GM) detectors:** gas-filled tube detecting charged particles for rapid dose rate screening, surface contamination survey
- **Ionisation:** gas-filled chambers measuring radiation-induced ion pair generation for more precise dose rate measurements

- **Gas proportional counting:** gas multiplication for enhanced signal detection, used for surface contamination measurements from alpha-, beta- and gamma-emitters
- **Gamma spectrometry:** semiconductor detectors that provide high-resolution gamma-ray spectroscopy, allowing identification of specific gamma-emitting radionuclides by their characteristic energy peaks
- **Liquid scintillation counting:** Systems that mix the sample with a scintillation cocktail to detect light pulses produced by alpha and beta radiation, particularly effective for low-energy beta emitters
- **Alpha-Induced Radioluminescence Imaging:** detecting light produced by alpha particles in air for alpha contamination detection during surface contamination mapping
- **Alpha spectrometry:** silicon semiconductor detectors in vacuum chambers with multi-channel analysers that measure the energy of alpha particles after radiochemical separation of samples
- **Mass spectrometry:** analytical technique that separates ions based on their mass-to-charge ratio (used for long-lived isotopes with low specific activity)
- **Passive/Active Neutron interrogation systems:** techniques that measure neutron emissions or induced fission neutrons to characterise nuclear materials in waste packages (used for neutron-emitting material quantification e.g. fissile)

Specialised measurement systems such as Segmented Gamma Scanning (SGS) and Tomographic Gamma Scanning (TGS) enable detailed characterisation of waste packages [6]. These systems provide vertical activity profiles or three-dimensional activity distribution maps, particularly valuable for heterogeneous waste assessment.

A critical consideration in radiological characterisation is accounting for matrix effects that can significantly impact measurement accuracy. Techniques such as transmission measurements with external sources and density correction factors help address these challenges.

## 2.4 NDT vs DT Application Necessity

Radioactive waste characterisation employs both NDT and DT, each with distinct advantages and limitations. Selection between these approaches depends on waste characteristics, information requirements, and practical constraints.

### 2.4.1 NDT

NDT analyse waste packages without sampling or altering their physical integrity:

- **Radiation measurements:** Gamma spectrometry, neutron counting, dose rate assessment
- **Imaging techniques:** Radiography, tomography, ultrasonic inspection
- **Physical property measurements:** Weight, dimensions, external condition assessment

Table 1 – NDT Advantages and Limitations

Advantages:	Limitations:
Preserves waste package integrity	Limited sensitivity for alpha and beta emitters
Reduces personnel exposure	Interference from dominant gamma emitters masking others

<b>Assesses entire waste packages rather than samples</b>	Attenuation effects in dense or heterogeneous matrices
<b>Minimises secondary waste generation</b>	Difficulty detecting chemical constituents
<b>Enables automated, high-throughput measurements</b>	

#### 2.4.2 DT

DT involve sampling waste materials for laboratory analysis:

- **Sampling and radiochemical analysis:** Collection and processing followed by chemical separation and measurement
- **Chemical analytical methods:** Dissolution, extraction, or other preparation steps
- **Material property testing:** Tests that may consume or alter specimens

Table 2 – DT Advantages and Limitations

<b>Advantages:</b>	<b>Limitations:</b>
<b>Higher sensitivity for difficult-to-measure radionuclides</b>	Generates secondary waste
<b>More complete chemical characterisation</b>	Increases potential personnel exposure
<b>Better discrimination between similar radionuclides</b>	Raises representativeness concerns for heterogeneous waste
<b>Direct measurement of specific physical properties</b>	Time and resource (skill and cost) intensive
	Challenging for packaged waste

Optimal characterisation strategies typically integrate both approaches, with NDT serving as the primary method and DT providing calibration, validation, or supplementary data [17]. Common integration strategies include:

- Using DT to establish correlations (SF) applied with NDT for routine characterisation
- Verifying NDT results through periodic DT analysis of representative samples
- Applying NDT for screening and DT for focused investigation of identified issues

IAEA guidance recommends a graded approach to characterisation, with the extent and type proportional to the potential hazard and intended management route [8].

## 2.5 DTM analysis and SF

### 2.5.1 Challenges in measuring difficult-to-measure radionuclides

DTM radionuclides include pure beta emitters, alpha emitters, low-energy gamma emitters. Direct measurement typically requires sampling, chemical separation, specialised measurement techniques,



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and complex data analysis. These processes are time-consuming, labour-intensive, and generate secondary waste, making comprehensive direct DTM analysis impractical for large waste volumes.

### 2.5.2 The SF Methodology

The SF methodology provides a practical site- and waste-specific approach to estimate DTM radionuclides activities without extensive direct measurements [18]. This approach establishes correlations between easily measurable key nuclides (KN) and DTM radionuclides.

The SF is defined as:

$SF = \text{Concentration of DTM radionuclide} / \text{Concentration of key nuclide}$

Once established, SF allow estimation of DTM radionuclide activities by measuring only the key nuclide activity and applying the appropriate factor. Key nuclides are typically gamma-emitting radionuclides (e.g.,  $^{60}\text{Co}$ ,  $^{137}\text{Cs}$ ) that are easily measurable and maintain consistent relationships with associated DTM radionuclides.

Implementation involves:

- **Historical knowledge collection:** Gathering information on waste origin and processing
- **Sampling and analysis:** Obtaining representative samples for comprehensive radiochemical analysis
- **Statistical analysis:** Determining SF through correlation analysis
- **Validation:** Confirming reliability through additional measurements
- **Implementation:** Applying validated SFs with key nuclide measurements
- **Periodic verification:** Updating SFs when processes change

Statistical considerations are crucial in SF methodology, including correlation analysis, appropriate data transformation (typically logarithmic), uncertainty quantification, and outlier management [13]. Recent advances include Bayesian approaches that allow continual updating of SFs as new data become available [19].

While widely applied, SF methodology has limitations including waste stream specificity, temporal variations in radionuclide relationships, and matrix effects. Verification through periodic direct measurements, statistical process control, and theoretical model comparison helps address these limitations.

### 3. NDT

This section focuses on NDT specifically applicable to large-volume radioactive waste characterisation. Rather than providing an exhaustive overview of all available non-destructive methods, we concentrate on technologies that have demonstrated practical utility for industrial-scale waste characterisation during decommissioning activities.

Many advanced NDT originally developed for security applications, such as border control and cargo inspection, have been successfully adapted for radioactive waste characterisation. These cross-domain technologies offer particular advantages when dealing with large, dense waste packages where traditional characterisation methods face significant limitations. The methodologies discussed emphasise approaches capable of penetrating shielding materials, identifying heterogeneities, and providing accurate activity distribution data for high-density waste forms commonly encountered in decommissioning projects.

The adapted technologies include advanced radiation-based imaging systems, neutron interrogation techniques, and hybrid methodologies that combine multiple physical principles to overcome the challenges presented by complex waste packages. Special attention is given to detection sensitivity, measurement speed, geometry flexibility, and data integration capabilities – all critical factors when characterising large waste volumes with potentially heterogeneous activity distributions.

#### 3.1 Physical properties

This section focuses on characterisation NDT aimed at reconstructing physical properties of the investigated radioactive waste, e.g. the waste matrix materials and density spatial distribution, the presence of voids and liquids, and other features expected to be determined in WAC.

These methods rely on physical interactions like sound, calorimetry, and radiation-based testing to detect defects or evaluate properties.

##### 3.1.1 Visual testing

One of the non-destructive assay methods for determining physical parameters is the visual examination of the waste matrix surface or the waste container. Visual examination can be used as one of the methods to examine the nature of the corrosion processes on the surface of the matrix or containers that may have occurred during the storage of packages. Cemented blocks – if prepared properly – have a surface without any cracks immediately after the immobilisation of waste. Over time, due to temporal changes in the cement matrix, seasonal temperature fluctuations, or, less frequently, as a result of radiolytic gas release and the possible generation of small particles, the surface of the matrix may change. These factors can also affect the shape and structural integrity of the waste package. This damage can be observed through visual examination. However, to detect cracks and defects within the depth of the block, other methods are required [20].

Visual examination is used to monitor the condition of waste packages during storage. Remote visual inspection, in particular, using optical tools such as telescopes, borescopes, fibre optics, and cameras, can be used for the remote examination of waste that emits significant radiation fields.

##### 3.1.2 Acoustic emission

Acoustic emission (AE) detection utilises specialised high-sensitivity transducers to capture elastic stress waves generated when materials undergo microstructural alterations. When cemented radioactive waste forms develop microfractures or experience internal degradation, these waves propagate through the material and are converted into analysable electrical signals. The technology functions within a frequency range of 20 kHz to 1.2 MHz [21], enabling real-time, passive monitoring of structural integrity without external material stimulation. This detection method spans multiple scales of phenomena – from large seismic events down to microscopic defect movements measuring mere picometers [21] in stressed materials. Various processes generate detectable acoustic signatures,



including cement hydration reactions [22], electrochemical degradation like metal corrosion [23], and mechanical responses such as crack formation and propagation under different loading conditions [21],[24]. The non-destructive nature of acoustic emission monitoring provides significant advantages for radioactive waste management by enabling early identification of potential structural issues before visible damage occurs. This capability allows for pre-emptive intervention, substantially enhancing containment safety protocols and minimising release risks. The continuous surveillance capability functions effectively in both laboratory testing environments and actual storage facilities, creating a robust monitoring solution throughout the lifecycle of cemented waste forms. The technique's sensitivity to microscopic changes offers unprecedented insight into material behaviour, with detailed classifications of acoustic emission sources across different materials thoroughly documented in comprehensive reference works [21],[24].

### 3.1.3 Ultrasonic testing (UT)

The ultrasonic testing method involves the use of ultrasonic pulses to check the internal homogeneity of the waste matrix and the container materials. Ultrasonic generators produce pulses that pass through the material, while receivers detect the reflected signals to identify internal defects or inhomogeneities.

Conventional Ultrasonic Testing: High-frequency sound waves are used to penetrate the material, enabling the detection of internal defects, thickness measurements, and the assessment of structural homogeneity.

Air-Coupled Ultrasonic Testing (Air-Coupled UT): Non-contact air-coupled ultrasonic transducers can provide circumferential measurements to detect swelling in drums and offer screening for discontinuity defects such as cracks or corrosion cavities [25].

Phased Array Ultrasonic Testing (Phased Array UT): This modern technology utilises multi-element sensors to produce detailed images of internal defects within the material structure [26].

### 3.1.4 Thermography

Temperature as a NDT like heat maps can add surface-based information and measuring temperature by heat generation inside a package can add content-based information. Compared to more common transmission based NDT, temperature information needs more context to be related to internal composition of a package. Heat maps can reveal inconsistency and total temperature generation might be related to chemical processes or radiological events [27].

### 3.1.5 Liquid penetrant testing

Liquid Penetrant Testing (PT) works by applying coloured dye or fluorescent liquid to surfaces. The liquid seeps into surface-breaking discontinuities through capillary action and remains there after carefully cleaning the surface. When a developer is applied, the trapped liquid is drawn out, creating visible indications of surface flaws.

The process typically follows five key steps: surface preparation, penetrant application, excess penetrant removal, developer application, and inspection/evaluation. Two main types exist: visible dye penetrants that can be inspected in normal lighting, and fluorescent penetrants that require ultraviolet light for inspection, offering enhanced sensitivity.

This method provides detection of surface-breaking discontinuities as small as 20-30 micrometers in width on materials with non-porous surfaces and is particularly effective on metals, plastics, ceramics, and glass, but cannot detect subsurface defects. The method is governed by standards such as [28], which provide guidelines for proper application and interpretation of results. Indications are evaluated based on their size, shape, location, and quantity to determine the severity and nature of defects.

## 3.2 Radiation-based testing

### 3.2.1 Transmission/Scatter testing 2D & 3D

Radiation-based testing (RT) is a NDT that uses radiation to fully penetrate the object from one side and detection and visualisation of the resulting radiation at the other side (transmission). The resulting detection at the other side will relate to interaction with the transmitted radiation and the material composition of the object. Most common examples are X-ray inspection like weld inspections, and luggage checks at airports that are a result in general absorption differences, hence density distributions. Radiation sources other than X-ray machines to create higher penetration energies can be linear accelerators (LINAC), industrial radioisotopes, neutron generators, up to cosmic rays and use muons as transmission source. Lower energy X-ray, gamma-rays and radiowaves can be used to analyse scattered radiation after partly penetrated, like ground penetrating radar (GPR) and X-ray fluorescence analysis (XRF). Detected radiation can be processed on a data analysis level, into a two-dimensional visual presentation, a three-dimensional reconstruction and in time (Realtime or monitoring over time periods), depending on the setup of source, object and detection methods.

### 3.2.2 2D-X-ray-radiography

The simplest and fastest inspection methodology to detect internal difference in densities, and the presence of liquids is 2D-X-ray radiography. 2D uses X, gamma rays or neutron to produce images of internal structures and detect voids, cracks, or inclusions. It is suitable for light packages, not for heavy iron or concrete containing packages. Furthermore, 2D images cannot provide the exact position of point of interest.

### 3.2.3 Gamma inspection and transmission tomography

Gamma rays can overcome the limited penetrating capacity of X-rays, inspecting the package more deeply with several methodologies according to the complexity of the package material matrix. Heterogeneous materials and densities spatial distribution requires the execution of tomographic methods exploiting specialised HPGe detectors and protocols. Since homogeneous materials and densities spatial distribution may simplify the techniques, studying the package by homogenisation (e.g. while in rotation) is the best solution when the object of characterisation is movable. The implementation of such technique requires the availability of a radioactive source to test the package (usually > 100 MBq), and a fine-tuned 4-axes mechanical system (in a laboratory environment usually) [29].

### 3.2.4 Neutron inspection

Neutrons are particularly sensitive to light materials (water, paraffin wax, etc.) or can stimulate prompt neutron-reactions with the emission of secondary gamma radiation by means it is possible to identify material composition and densities. Such methods use fast neutrons sources (radioactive sources, neutron generator, or accelerator, with neutron yields higher than  $10^8$  n/s) and they usually require proper shielding to be executed safely.

### 3.2.5 Accelerator-based inspection

They provide a combination of gamma and neutron techniques to test the package. Accelerators may have the ability to test particularly heavy packages, with massive shielding due to the higher penetrating capacity of gamma and neutron radiation here produced. Accelerators may allow to put in place target-particles techniques also. The drawback is that such high penetrating radiation may require massive concrete shielding materials to be executed safely making such techniques almost fixed installation.

### 3.2.6 Cosmic radiation-based inspection (muon tomography)

Using cosmic radiation as a natural occurring transmission source, the muons have a high penetration power, but the direction, orientation and flux is fixed. This NDT is still under development and applied as the only option for objects as large as the pyramids, volcanoes and underground cave systems and

explorative also on nuclear waste container concepts [30]. This novel technique is not practical to setup due to the measurement area detection before and after transmission to the object, the long exposure times needed for resolution and contrast in density. The results thus far are promising, and numerous potential applications have been identified by the IAEA [31].

### 3.2.7 Scatter based inspection

Ground penetrating radar (GPR) assesses the condition of concrete structures by identifying rebar, conduits, and potential deterioration within the material. The technology offers a trade-off between detail and depth: higher frequency antennas provide finer resolution but shallower penetration, while lower frequency antennas can reach greater depths with reduced detail resolution.

Typically, GPR systems can penetrate up to 100 feet (30 meters) in ideal conditions, though actual performance varies significantly based on environmental factors and material composition [32].

### 3.2.8 Synchrotron X-ray characterisation techniques

Modern synchrotron-based spectroscopic and scattering techniques offer the opportunity to probe interface and surface structures down to the atomic length scale and gain data of exceptional quality for structural studies. Especially spatially resolved X-ray Absorption Spectroscopy (XAS) and X-ray Diffraction (XRD) investigations (micro-XAS/XRD) combined with micro-XRF allow to gain spatially resolved micro-scale information to pin down the influence of the heterogeneity of the complex alkali-silica reaction (ASR) system [33]. The synergistic use of micro-XRF with micro-XRD opens the possibility of determining the element distributions in the complex ASR zones and subsequent the structural refinement of crystalline ASR phases.

Recently, synchrotron-based microspectroscopic investigations revealed the nature of the crystalline and amorphous phases formed in micro-cracks of concrete aggregates as a consequence of ASR [34],[35],[36],[37],[38].

In addition, scanning transmission X-ray microscopy (STXM) [33] can be applied, which offer spatial resolution down to 20x20 nm<sup>2</sup>. With STXM investigations chemical maps at selected energies can be collected and it can be combined with near-edge X-ray absorption fine structure (NEXAFS) spectroscopy. Both XAS and STXM/NEXAFS allow to collect experimental spectra of elements of interest, which can be compared with spectra of adequate reference compounds, i.e., characteristic fingerprints in the spectra can be exploited. Coupled with state-of-the-art ab initio methods, such as FEFF [39] or FDMNES [49] the local structure (bond distance, coordination numbers and type of near neighbours) around an X-ray adsorbing atom in a crystalline or amorphous structure, can be determined from XAS and NEXAFS spectra [50].

## 3.3 Chemical properties

Non-destructive measurement of chemical properties of large waste volumes is mostly related to waste acceptance criteria, for example safety during transport and storage and long-term stability, integrity to the containment. Some chemical related problems have only occurred after long time storage, for example corrosion, gas build-up and gel forming, thereby increasing the waste acceptance criteria over time. Although the focus is on non-destructive investigations, chemical properties are mostly examined in-directly via a fraction by sampling or indirect via the equilibrium with the environment. Sampling can be done strategically to be representative of the waste, in time before storage or enclosure and in context of known waste streams, thus one sample for several volumes. The listed techniques are therefore sample based or monitoring based, for example attached to the raw waste, enclosure and environment. The need to innovate a technique from sample analysis into more closely positioned to the real waste, is not yet explored.

**Corrosion Mapping** uses electrochemical interactions to evaluate surface corrosion. By measuring electrical properties across a material's surface, technicians can create maps showing where and how severely corrosion has affected the material.

**Chemical Composition Analysis** identifies what materials are made of and detects impurities or inconsistencies. While this technique may remove microscopic amounts of material for testing, it's still considered NDT because the impact is minimal and does not affect structural integrity.

These methods provide valuable information about material quality and condition without compromising their functional properties.

### 3.4 Radiological properties

The hazard of radioactive waste depends on the content of the radionuclides and their concentration, i. e., on radioactive properties in it. A waste with activity concentrations equal to, or less than, clearance levels is considered nonradioactive. That is why the exact radioactive content in every radioactive waste should be determined. The different methods of measurements have been developed for screening the waste composition.

Besides the increase in technology to measure radiological properties more accurate, faster and extend the possibilities to measure more different radio nuclides, the measurement of dose rate and contamination are more focused on speed, ease of use and robustness. Mandatory dose rate declaration on package surface in one meter distance and alpha/beta count rate on swipe samples are not radio nuclide specific. The equipment is chosen due to safety related regulatory standards and not added value to understand the waste composition. The same safety related dose rate and contamination risks are addressed for checking clean work space [40].

As a routine, it is envisaged to first measure dose rate at the sampling place and after to take a smear from the measured surfaces. It is supposed that dose rate value correlates with a contamination level. Sampling by taking a scraper for destructive sample analysis is usually performed in the places with a highest dose rate on various types of material where is anticipated a high level of activation/contamination of the material, but also sampling is performed in the places of a lower dose rate to enable creation of reliable scaling factor of dedicated waste stream.

The listed techniques next are focused on radiological waste declaration starting from gross measurements to accurate non-destructive measurement technique.

#### 3.4.1 Dose rate measurements

For the general radiation surveys, contamination monitoring, ensuring workplace safety or determination of representative sampling collection places, the results of dose rate measurements and alpha and beta/gamma contamination counters can be used. Dose rate measurement is the simplest and cheapest contaminated areas / radioactive waste characterisation method. These measurements are typically performed using instruments such as ionisation chambers (for high-dose rate environments), GM counters (for beta and gamma radiation), and scintillation detectors (for gamma and neutron radiation).

##### **GM detectors**

Measurement equipment containing GM detectors are used to determine external radiation exposure, by continuously measuring the ambient gamma dose rate. The GM Counters provides rapid and reliable detection of small changes in environmental radioactivity over a large area, as well as the identification of any increase resulting from nuclear activities. GM detectors can measure over a wide range of 0.05-10 Sv/h of dose rate. While GM counters are popular for their simplicity and cost-effectiveness, they have limitations, such as the inability to differentiate between radiation types and reduced accuracy at high radiation rates due to "dead time". To address these issues, modern GM counters incorporate compensation circuitry to correct for dead time, improving their reliability in various applications. Summarising, GM survey meters are radiation detectors used to detect radiation or to monitor for radioactive contamination. GM detectors usually have a window either at the end or on the side of the detector to allow alpha or beta particles to enter the detector. These detectors may have a variety of

window thicknesses, however, if the radiation cannot penetrate the window it will not be detected. Depending upon the window thickness, GM systems can detect X-ray, gamma, alpha, and/or beta radiation. Radioactive materials that emit these types of radiation (e.g.  $^{14}\text{C}$ ,  $^{22}\text{Na}$ ,  $^{32}\text{P}$ ,  $^{35}\text{S}$ ,  $^{45}\text{Ca}$ ,  $^{51}\text{Cr}$ ,  $^{60}\text{Co}$ ,  $^{137}\text{Cs}$ ) can usually be detected using GM survey meters. Because appropriately configured GM detectors are more sensitive to X-rays, gamma-rays, and high energy beta particles and less sensitive to low energy beta and alpha particles, they are usually not used to detect alpha or very low energy beta particles. Thus, GM tubes are not useful for monitoring  $^3\text{H}$  or  $^{63}\text{Ni}$ , nor are they sensitive enough to detect very small amounts ( $< 37 \text{ Bq}$ ) of low energy beta or gamma emitting radionuclides such as  $^{14}\text{C}$  or  $^{125}\text{I}$  [41].

### **Ionisation chambers**

Ionisation chambers are measurement standard for high activity sources such as radiation hot cells or medical equipment (dosages of radiopharmaceuticals and X-ray / radiotherapy exposure) survey purpose, as they can tolerate prolonged periods in high radiation fields without degradation. These meters provide accurate dose rate readings, works well for high-energy gamma radiation, less sensitive to low-energy radiation and requires calibration. Recent advancements in ionisation chambers focus on enhancing their accuracy and durability.

### **Gas proportional counters**

Gas proportional counters are used for gamma dose rate measurements with their high efficiency for gamma and also adaptiveness to low-energy beta and alpha particles due to good discrimination between alpha and beta radiation.

### **Scintillation Detectors**

Scintillation detectors are specialised measurement tools for radiation surveys, contamination monitoring, and workplace safety assessments, particularly effective for detecting gamma and neutron radiation across various environments. These detectors utilise specialised crystals that convert radiation into light pulses, which are then amplified by photomultiplier tubes to provide detailed radiation measurements. They excel in general radiation characterisation, demonstrating high sensitivity for gamma radiation and offering capabilities for neutron detection through specific converter materials. While providing cost-effective and relatively simple measurement techniques, scintillation detectors require careful calibration and are most effective in moderate radiation fields. [44]

Dose rate measurement is a useful method for the preliminary determination of the homogeneity of the waste flow to enable the definition of the sampling strategy, including the requirements of worker protection. Dose rate measurements can be sufficient to confirm the radiological characteristics of stable waste streams if there is supportive evidence of their composition and stability. For example, for wastes that contain a single gamma emitting nuclide, dose rate or gross gamma measurements are usually sufficient to characterise the radioactive properties. The other examples of the wastes, where dose measurements can be applied for characterisation include the following:

- **Spent sources:** constituents will be well known, and detailed documentation will be available
- **Enrichment, conversion and fuel fabrication:** The radioactive species will only be the fissile material. The nuclide vector will be known, as this is a highly controlled part of the process. The significant process control effort required for manufacturing will be a valuable source of waste characterisation information. As the process is highly controlled, the streams will be stable for each particular batch
- **Institutional and radio-pharmaceutical wastes:** Similar to fuel manufacture, these processes tend to be highly controlled, with very few species. Process control data will be highly valuable to characterise wastes. Waste will be stable within batches



- **Spent fuel:** Spent fuel will not change in composition until it is reprocessed. Although nuclide ratios will change due to decay, this is a very well-understood process and can be predicted very accurately [42].

### 3.4.2 Surface contamination measurements

Surface contamination measurements detect loose and fixed radioactive contamination on waste surfaces. Common instruments for in situ total (loose and fixed) surface contamination measurements include alpha/beta proportional counters (for distinguishing alpha and beta emitters), liquid scintillation counters (for detecting low-energy beta emitters like tritium), GM counters with thin-window detectors (for beta and some alpha measurements). Laboratory determination of loose surface contamination requires using smear tests and swipe sampling. Surface contamination measurements are applied in waste segregation when it is important to distinguish between clean and contaminated materials, to differentiate between surface and volume contaminated waste, to prevent contamination spread in storage and handling areas, and to determine if waste requires decontamination before disposal or transport [42].

#### Gas proportional counters

As it was mentioned above, the proportional counter can be set to reject pulses below a given size by use of bias levels or sensitivity settings making it easy to count for  $\alpha$ -particles only in a mixed  $\alpha/\beta$  sample either by lowering the high voltage to the  $\alpha$ -plateau level or only counting pulses above a certain energy level. Similarly, one can count only smaller  $\beta$  pulses by not allowing large pulses to be counted. The chamber may be either windowless or have a very thin window (e.g., 0.9 mg/cm<sup>2</sup>). The chamber is made from high Z material to shield against gamma and background with gas inlet and outlet ports to allow gas to flow through chamber. The filling gas may flow continuously during the counting cycle or may only purge the chamber after each count. The gas normally used for mixed  $\alpha/\beta$  samples is P-10 gas, consisting of 10% methane and 90% argon; however, pure argon may be used for analysing samples emitting only  $\alpha$  particles. Proportional counters are simple pulse counting devices versus exposure measuring instruments like ion chambers. They are used primarily in the laboratory for beta, alpha, and neutron detection in which a special chamber is required for neutron detection because of the need to moderate and then capture the neutrons and subsequently count the resultant radiation. At one time portable proportional counters were employed and some (windowless) detectors were fabricated for tritium detection. While these may still be used in some facilities, LSC counting is by far more sensitive in checking for removable contamination. In laboratory counting, because there is a minimum sample to window distance, or perhaps a windowless configuration, the sample is practically in intimate contact with sensitive volume. Some sample self-absorption may occur so the maximum sample thickness should be between 1.2 – 0.6cm to allow all particulate events to have a good probability of being counted. Most systems are  $2\pi$ , that is the sensitive volume forms a hemispherical dome around the sample. Therefore, the maximum efficiency is about 50%. However,  $4\pi$  systems are available with ultra-thin windows. Given this geometry, the intrinsic efficiency is greater than 99% for alphas and betas which can pass through the window. Some typical efficiencies to be expected are: <sup>14</sup>C - 40%, <sup>90</sup>Sr - 55%, <sup>210</sup>Po - 35%, and for gamma rays- 0.5 - 1% for 0.1 to 2 MeV [41] As an example, thermal oxidation combined with the gas detector technique has been used for the <sup>14</sup>C concentration determination in irradiated graphite from the Oldbury reactor [41]. In-situ the total  $\alpha$  and  $\beta/\gamma$  surface contamination can be performed with the portable device (Thermo Scientific™ FHT 111 CONTAMAT Contamination Monitor [43]) on surfaces of the reactor constructions to determine the type of contamination.

#### Scintillation counters

Organic scintillators with appropriate modifications can substitute the other detectors for different applications as medical imaging, nuclear plant safety, and homeland security. Polyethylene 2,6-naphthalate (PEN) is perspective scintillator, because it has very high stability, easy to produce in big quantities and can have big surface area, it could be used to distinguish all kinds of ionising radiation

with possibility to analyze their spectral characteristics [44]. ZnS(Ag) based detectors are used for alpha particles [45]. Scintillation detectors convert radiation into light, which is then detected by a photomultiplier tube, usually it is high sensitivity and energy resolution, can differentiate between different radiation types and energies.

### **Alpha-induced radioluminescence imaging**

A significant advancement in remote alpha radiation detection is the use of alpha-induced radioluminescence imaging. This technique detects photons emitted from nitrogen molecules excited by alpha particles, enabling remote imaging of alpha emitters with high sensitivity and spatial accuracy. In [46] it was demonstrated the detection of a 29 kBq  $^{241}\text{Am}$  source at a distance of 3 meters within 10 minutes, highlighting the potential of this method for safer and more efficient alpha monitoring in nuclear forensics and waste management. The idea is based on nitrogen molecules excitation by alpha particles which ionise molecules via secondary electrons, which in turn, excite the surrounding nitrogen molecules and returning to their ground state, they emit UV photons. These photons can be detected via charge-coupled device cameras provided with the sandwich structure of different filters achieving measurable optical density at the room light background (OD11).

### 3.4.3 Active and passive neutron interrogation for nuclear characterisation

Radioactive Waste Package (RWP) characterisation is a complex and critical task involving the qualification and quantification of the radiological content of nuclear waste. This includes dose rate measurements, spectroscopy, isotopic composition analysis, and more. NDA methods are crucial as they minimise radiation exposure to personnel, prevent the production of secondary radioactive waste, reduce costs, and provide a comprehensive characterisation of waste packages within reasonable measurement times.

Active and passive neutron techniques are essential in the non-destructive analysis of nuclear materials, specifically in the characterisation of RWP. These techniques are fundamental; they offer a solution for managing radioactive waste, ensuring that nuclear materials are handled safely and efficiently based on non-destructive radiologic measurements, especially with waste packages that must be verified for the presence of fissile or fertile materials.

**Passive neutron techniques:** Neutrons emerging from the package can be used to detect the presence of radionuclides decaying by spontaneous fission. Fission neutrons are emitted “packed-in-time” so, if neutrons are detected on a sharp-tuned time scale, fission events can be counted and the equivalent mass of  $^{240}\text{Pu}$  can be estimated under certain conditions. It is the case of the Neutron Coincidence Counting techniques or Multiplicity Counting.

Passive Neutron Coincidence Counting (PNCC) detects neutrons emitted from spontaneous fissions of isotopes like  $^{240}\text{Pu}$ . This method is advantageous for non-intrusive measurements but is susceptible to matrix effects, where surrounding materials can alter neutron behaviour and, as a consequence, their detection. This technique is particularly useful in identifying and quantifying actinides, primarily plutonium, within medium-sized waste drums, better if filled with metallic waste than concrete matrix [47].

**Active neutron techniques:** Production of neutrons by fissions on fissile materials eventually inside the package can be stimulated by “external” neutrons, so that the execution of active neutron methodologies is exploiting the introduction of an external neutron source (radionuclide-based or accelerator-based) to test the package. Neutrons emerging from the package are usually detected on a sharp-tuned time scale, and several analysis methodologies can be put in place (Coincidence Counting or Differential Die-Away time techniques). The final output is the counting of fission events, and the equivalent mass of a selected reference fissile element ( $^{235}\text{U}$  or  $^{239}\text{Pu}$ ) can be estimated under certain conditions. Active Neutron Interrogation (ANI) employs external neutron sources (e.g.,  $^{252}\text{Cf}$  or neutron generators) to induce fission in target materials like  $^{235}\text{U}$  or  $^{239}\text{Pu}$ . This method excels in detecting fissile material in samples where passive methods are insufficient [48].

The MICADO project [11] has significantly advanced neutron interrogation technologies by developing a modular, transportable neutron measurement system based on  $^3\text{He}$  detectors. This system integrates high-efficiency neutron detection arrays, Monte Carlo (MCNP) simulations to model and refine neutron behaviour, and Artificial Neural Networks (ANNs) to correct for matrix effects, enhancing the accuracy of nuclear mass estimations. Experiments conducted at CEA Cadarache [51] have validated the system's efficiency, showing deviations within 10-20% between estimated and actual plutonium mass, except for highly moderating matrices like polyethylene. Additionally, ANI has demonstrated promising results, with mass estimates deviating within 15-40%, except for materials with unknown compositions like PVC, thanks to the introduction of ANN technique based on the study of matrix material effects on measurements. The MDA quantifies the lowest activity level detectable with statistical confidence. For MICADO, detection limits for  $^{240}\text{Pu}_{\text{eq}}$  and  $^{239}\text{Pu}_{\text{eq}}$  in passive and active modes are below 1 g for a 30-minute measurement, making it a robust tool for legacy waste assessment.

The combination of active and passive neutron techniques, supported by advanced computational models and machine learning, represents a cutting-edge approach to nuclear material characterisation. These methodologies not only enhance the accuracy and efficiency of nuclear waste management but also ensure alignment with rigorous regulatory standards. By integrating these advanced techniques, these projects provide valuable contributions to the field of nuclear waste management, promoting safer and more effective practices across the industry.

#### 3.4.4 Gamma spectrometry

The characterisation of radioactive waste is an essential component of waste management and safeguards in all nuclear sectors (fuel cycle, decommissioning, dismantling, medical field, etc.). To determine the classification of the RWP, a non-destructive characterisation of the RWP content is required. In order to perform this characterisation, the usual technique used is gamma spectrometry measurement. It exploits the emissions of characteristic X-ray and gamma radiation from radionuclides, providing a non-destructive means to both identify and quantify these substances based on their specific energy signatures and intensities [52].

##### 3.4.4.1 Applications in decommissioning and regulation compliance

**Decommissioning waste characterisation:** In the context of nuclear power plant dismantlement, gamma spectrometry is indispensable for categorising waste according to its radiological content, aiding in the efficient segregation and management of radioactive materials.

**Waste package verification:** This technique ensures that all stored or disposed waste complies with stringent regulatory frameworks, safeguarding public health and environmental standards.

**Fissile material monitoring:** Gamma spectrometry plays a pivotal role in the identification of materials that pose proliferation risks, thus contributing to global nuclear non-proliferation efforts. Due to the low intensities and energies of gamma rays produced by U and Pu, fissile material monitoring through gamma spectrometry it is not a suitable solution especially for dense matrix or large packages. The only possibility to have information about U and Pu content through NDT is by the combination of the analysis on the eventually detectable gamma radiation and active/passive neutron techniques.

##### 3.4.4.2 Advanced measurement techniques in gamma spectrometry

Non-destructive gamma spectrometry is a cornerstone for assessing RWP, determining the presence and quantifying the Minimum Detectable Activity (MDA) of radionuclides. This is accomplished through various specialised measurement strategies:

**Open geometry:** often applied to a diverse array of container types—from small polyethylene bins to large concrete casks—this technique can be constrained by radiation attenuation within dense materials, necessitating the segmentation of concrete waste for effective measurement in volumetric containers.

**Segmented measurements and emission tomography:** this method enhances the specificity of activity determination within waste containers. By employing a collimator to narrow the detector's field



of view, the system conducts detailed scans through translational, rotational, or elevational movements of either the package or the detector. This technique includes gamma scanning for complete slice measurements and emission tomography for detailed sectional activity distribution, utilising angular scans to enable three-dimensional reconstructions of waste activity. Homogeneity or heterogeneity of radioactive material spatial distribution drives the complexity of the technique to be applied, ranging from the simplest possibility, the Open Geometry (a fixed “radiological snapshot”), passing through Angular Scanning, to the more complex emission tomography providing the 3D reconstruction of radioactive material spatial distribution within the inner matrix of the package. The implementation of the emission tomography technique requires a fine-tuned 4-axes mechanical system (in a laboratory environment usually) and it may require the previous execution of a transmission tomography [53], [54].

**Fixed measurement:** critical for the assessment of large stationary objects within nuclear decommissioning projects, such as reactor vessels or steam generators, if calibration hypotheses meet the real scenario, this approach allows for precise activity distribution mapping, which is essential for appropriate waste classification and disposal.

#### 3.4.4.3 Technological Composition of gamma measurement stations

A typical gamma spectrometry station comprises a detector, coupled with either analogue or digital electronic systems, and a comprehensive analysis suite. The integration of these components often permits remote control operations, enhancing the precision and safety of measurements. Selecting the appropriate measurement type and detector technology is crucial for meeting both regulatory and operational demands. The predominant detectors employed include:

**High-Purity Germanium (HPGe) detectors:** reference in the field, known for their excellent energy resolution, these detectors are considered the gold standard in gamma spectrometry, albeit requiring significant refrigeration.

**Inorganic and organic scintillators:** such as LaBr<sub>3</sub>, NaI, CsI, and plastic or liquid scintillators, which, despite their lower resolution, are valued for their large volume manufacturing capabilities and utility in less complex spectral analyses.

**Semiconductor detectors:** devices like CdTe or CdZnTe operate at room temperature and offer slightly superior resolution over scintillators but at a reduced detection efficiency.

#### 3.4.4.4 Gamma spectrometry in combination with Monte Carlo simulations

An intensive use of Monte Carlo simulations may be expected in combination with gamma spectrometry for efficiency calibration when experimental calibration is difficult to implement. In these cases, Monte Carlo simulations is the optimal solution for leading to more accurate radiological characterisations.

Other applications of Monte Carlo simulation in combination of gamma ray spectrometry have been tested, one of them has been reported below.

Regarding the waste of low activity, the aim of characterisation is to decide if decontamination will be efficient as well as to select the most effective decontamination techniques. The gamma spectrometry technique is used to distinguish activation from contamination on metallic components. Combination of gamma spectrometry measurements and Monte Carlo simulation allows distinguishing of surface contamination from volume activation by the shape/intensity and peak/Compton ratio of  $\gamma$ -spectra (of key nuclides <sup>137</sup>Cs and <sup>60</sup>Co) analysis of conventional HPGe, CeBr<sub>3</sub> or NaI detectors. This technique allows monitoring of metallic segments after dismantling and cutting, aiming at reduction of the measurement uncertainties related to the density and activity distribution. The method allows determination of activities of <sup>137</sup>Cs and <sup>60</sup>Co at the level of specific clearance for recycling in 1-2 min (amount of metal radioactive waste ~100 kg in each measurement) and allows reduction of uncertainties related to activity inhomogeneities by 30 %. This method aids to select the management route as well as the decontamination or clearance procedure. The technique was developed at TRL3 level.

In [57] a mock-up of an industrial measurement system for radioactive waste drums, containing bituminised radioactive sludge originating from the effluent treatment was developed to determine the radiological inventory of each waste package, with a special care to minimise the uncertainty on the total alpha activity by determining the low-energy photon attenuation in gamma-ray spectroscopy of bituminised radioactive waste drums using a peak-to-Compton ratio based on a ratio between the Compton continuum in a low-energy area and the main peak of the gamma spectrum.

#### 3.4.4.5 Incorporating AI and Machine Learning

Most of the innovation in the field is coming from the introduction of Artificial Intelligence (AI) and Machine Learning (ML) to enhance detection capabilities instead of focusing on detectors. These technologies facilitate automated spectrum deconvolution for improved radionuclide identification, real-time hotspot mapping to reconstruct activity distributions accurately, and adaptive background subtraction to adjust to dynamic radiation environments.

An example is what was done inside the MICADO [11] project on the gamma station. The project worked on different layers: upgrading the tomographic and SGS scanner in terms of detector, transmission source, and automation. It integrated two other detection systems: a gamma spectroscopic sensor on a robotic stand automatising all safety measurements and a fast hot spot search. The integration of a gamma camera for the visualisation of the presence of hot spots and first gamma identification. At the same time, a study and implementation of a procedure based on preliminary measurements taken by the gamma camera and the spectroscopic system to determine autonomously which measurement type between (segmented, angular, open geometry, or tomographic measurements) has to be performed. Finally, the connection of the gamma station to a software platform with all measurement reports stored in a database accessible from several users and finally a Monte Carlo analysis for the uncertainty reduction (pipeline assessment) of combined measurements for a single RWP (gamma and neutron).

#### 3.4.4.6 Accelerator-based techniques

Using electron accelerators combined with suitable targets they provide a combination of gamma and neutron techniques to inspect the package with active interrogation methods. Accelerators may have the ability to test particularly heavy packages, with massive shielding due to the higher penetrating capacity of Gamma (from Bremsstrahlung) and neutron radiation here produced. Fissile materials eventually contained can be stimulated with high energy Gamma rays that induce photofission, so that emerging neutrons can be counted and used to produce the estimation of the selected reference fissile element ( $^{235}\text{U}$  or  $^{239}\text{Pu}$ ). The drawback is that such high penetrating radiation may require massive concrete shielding materials to be executed safely making such techniques almost fixed installation.[55]  
[56]

## 4. Data management

### 4.1 Automated data collection and processing

Previous projects have advanced automated data collection and AI/ML-driven analytics for in-situ nuclear waste characterisation and decommissioning. In the PREDIS project, [58],[59] an Internet of Things (IoT)-based monitoring framework was developed for cemented radioactive waste, using Long Range Radio Wide Area Network (LoRaWAN), Hypertext Transfer Protocol Secure (HTTPS), Message Queuing Telemetry Transport Secure (MQTTs), and ontologies, such as Provenance (PROV-O) and Semantic Sensor Network (SSN), to standardise data provenance. The system processed real-time sensor data in Azure and InfluxDB, integrating ML models (TensorFlow, PyTorch, Statsmodels) for predictive maintenance, anomaly detection, and degradation forecasting. In the MICADO project [60],[61], the focus was put on automated radiological waste characterisation, using Radio Frequency Identification (RFID) tagging for real-time tracking and structuring data in the DigiWaste Database. Monte Carlo simulations validated sensor measurements against predictive models, improving uncertainty quantification and structured regulatory reporting. In the PLEIADES project [62],[63] an ontology-based decommissioning framework was introduced, integrating Building Information Modeling (BIM)-based digital models for waste classification, dose exposure assessment, and cost estimation. The platform facilitated scenario simulations and decision-making by aligning data with regulatory requirements, tested in real-world cases like Santa María de Garoña and Halden Research Reactor. In the CLEANDEM project [64],[65] an Unmanned Ground Vehicle (UGV) with integrated radiological sensors was deployed, transmitting gamma, neutron, and contamination data to a high-resolution Digital Twin (DTW) via the Qpro<sup>2</sup> Multiplatform. This enabled real-time radiation mapping, predictive decontamination planning, and risk assessment, improving efficiency in nuclear site remediation. In the INSIDER project [66],[67], the focus was put on in-situ radiological characterisation, implementing wired and wireless networks for data transmission and real-time monitoring in constrained environments. Emphasis was placed on uncertainty estimation methodologies to improve measurement assessments. Together, these projects have advanced data-driven nuclear waste management, integrating AI, new communication protocols, and ontology-based frameworks to improve automation, predictive modelling, and regulatory compliance.

### 4.2 Digital twin technology

Digitalisation and DTW are transforming RWM, providing enhanced monitoring, predictive modelling, and decision-making capabilities. DTW have been explored worldwide in various nuclear applications, from waste tracking and facility decommissioning to long-term safety assessments of geological repositories [68],[69],[70].

European projects have played a key role in advancing DTW applications in RWM. In the PREDIS project, a DTW framework was developed to simulate the long-term evolution of cemented radioactive waste packages, integrating physics-based models and machine learning for predictive modelling. The DTW incorporates chemical, mechanical, and environmental models to assess degradation mechanisms such as ASR, carbonation, and cement hydration. A prototype Jupyter-based dashboard enables interactive simulations, leveraging experimental data to refine predictions via Bayesian inference [71],[72],[73]. Future improvements target uncertainty quantification and real-time data integration for broader applicability in waste storage facilities. In the CLEANDEM project, a DTW system was designed and demonstrated at EUREX Saluggia for autonomous radiological monitoring and decision support in nuclear dismantling and decommissioning [7]. Integrated with UGVs and robotic arms, the DTW continuously updates 3D-mapped radiological data using gamma/neutron spectrometers, contamination detectors, and fibre-optic dosimetry. In the PLEIADES project, DTWs for nuclear decommissioning were foreseen, focusing on waste estimation and management through a BIM-based platform and a decommissioning ontology. The aim was to test DTW simulations for six use cases across three real-life cases in France, Norway, and Spain, covering cost planning, radiation exposure, and waste assessment through an iterative refinement [74].

These initiatives demonstrated the potential of DTWs to enhance efficiency, safety, and predictive capabilities in RWM.

### 4.3 Standardisation of data formats

RepMet (Radioactive Waste Repository Metadata Management) is an initiative by the OECD-NEA Integration Group for the Safety Case (IGSC) to standardise metadata management for radioactive waste repositories to ensure compatibility with international nuclear waste repositories and regulations. It defines structured metadata libraries for site characterisation, waste packages, and repository structures, ensuring data traceability, interoperability, and long-term usability across national programs [75],[76],[77]. RepMet promotes harmonised data formats, aligning with international nuclear waste management regulations and facilitating effective repository lifecycle documentation.

The PLEIADES project extended the OpenBIM standard from the building industry to nuclear decommissioning, enabling structured information exchange across waste disposal and dismantling activities [78], and PREDIS aligned with this approach by defining standardised input and output data formats for pre-disposal waste [79]. Additionally, the integration of BIM and GIS is recognised as a crucial step toward enhancing data traceability and interoperability in waste repositories [80]. Complementing these efforts, the Findable, Accessible, Interoperable, and Reusable (FAIR) principles are being adopted to promote data sharing, with European Open Science Cloud (EOSC) providing a framework for standardised data access and processing in nuclear waste digitalisation [80].

### 4.4 Blockchain-based data storage

Blockchain technology has emerged as a promising tool for enhancing traceability, security, and regulatory compliance in radioactive waste management [81],[85],[86]. Its ability to provide immutable records, decentralised data storage, and automated smart contracts make it ideal for ensuring waste lifecycle transparency. Research highlights blockchain's role in data integrity, access control, and systematic auditing, preventing tampering and unauthorised modifications. Proposed applications include blockchain-based radioactive waste tracking systems, integrating RFID, IoT sensors, and Global Positioning System (GPS) to monitor waste from generation to disposal [85],[87]. The IAEA has explored blockchain for secure digital tracking, improving safeguards and international regulatory compliance [88]. A DTW framework integrated with blockchain has been proposed to enhance RWM for storage facilities and disposal sites based on blockchain's immutable ledger to ensure data integrity, traceability, and secure access control while enabling real-time monitoring and predictive modelling through DTW technology [89]. As digitalisation advances, blockchain integration with DTs could enhance real-time waste monitoring, predictive modelling, and regulatory oversight.

## 5. DT

Characterisation of nuclear waste or nuclear waste packages is performed through both NDT and DT methods, allowing to determine their physical (density, volume, shape, position of the waste and embedding matrices, mechanical toughness, cracking, diffusion coefficient, gas release, thermal power, etc.), chemical (elemental composition, content of toxic or reactive substances, etc.) and radiological characteristics (dose rate,  $\alpha$  and  $\beta$  activity, isotopic composition and mass of nuclear materials, etc.) [90].

A significant limitation of NDT is that they generally measure properties at the surface of materials or waste, therefore care is needed to ensure that subsurface properties, including contamination, which may be shielded from detection, are not missed. Typically, the use of intrusive sampling and destructive analysis is required to give a full picture. Destructive analysis in a laboratory is likely to provide lower detection limits, more precise radionuclide measurements and can reveal subsurface properties of material or waste which are not seen when using NDT. Consequently, destructive analysis often constitutes a key aspect of characterisation allowing the development of SF (and radionuclide vectors) which underpins the wider use of inferred and NDT [91].

Therefore, DT are an essential complement to the NDT for the characterisation of radioactive waste, particularly for historic waste packages with little or insufficient available data [92].

Concerning radiological characterisation, DT and their full result analysis provide the most accurate and unbiased activity determination, since pure alpha and beta emitting radionuclides or those emitting gamma or X-rays with a too small intensity or energy are extremely difficult to measure in already conditioned waste packages [93].

Pure alpha emitters have extremely short ranges in matter – typically only a few micrometres in solids, making them completely undetectable from outside a waste package. Even thin packaging materials or matrix components fully attenuate these emissions. Similarly, beta-emitting radionuclides have limited penetration capabilities that prevent reliable external measurement in most waste matrices.

Some radionuclides that decay via electron capture emit only soft X-rays with energies below 10 keV. These low-energy emissions experience severe attenuation, with more than 99% being absorbed after passing through just a few millimetres of typical waste matrix materials. This makes them practically undetectable through non-destructive means, particularly in dense or heterogeneous packages. For this reason, alpha and beta emitters must be separated from the matrix to overcome self-absorption and allow their detection. Moreover, since the beta emission spectrum is continuous, the analyte of interest must be purified from other interfering radionuclides through radiochemical separations to allow for accurate measurement.

Destructive testing overcomes these limitations by:

- Completely dissolving or processing samples to eliminate matrix effects and self-attenuation
- Applying radiochemical separation techniques to isolate specific radionuclides from interfering elements and other radionuclides
- Preparing purified samples in optimal counting geometries for accurate measurement
- Utilising specialised detection systems calibrated for specific radionuclide types.

Destructive analysis typically involves sample destruction using acid digestions, oxidising agents and/or high temperature treatments. This generally results in the contaminants of interest being in a liquid form. Chemical separation processes can then be used to purify the required analyte or compound, which can then be analysed. For radiological characterisation typically the analyte of interest is prepared in the more suitable form (e.g. for alpha spectrometry evaporated or electroplated on to steel discs) which can then undergo radiation detection in a standard fixed geometry of known counting efficiency [91].



Radiometric determination is performed by instrumental analysis. Sophisticated methods are used such as liquid scintillation counters that allow beta detection, alpha spectrometry with semiconductor detectors, high resolution gamma spectrometry for high and low energy X- and gamma-emitting nuclides, mass spectrometry that gives an accurate and efficient response for the analysis of the prepared and/or separated waste samples [94].

Destructive characterisation also includes the measurement of non-radiological contaminants including physical; chemical and biological parameters which may be required to meet a range of characterisation objectives associated with decommissioning and waste management, including the protection of workers. Physical measurements may include as an example the shear stress of sludges or the grain size of solid materials. Destructive analysis may be used to determine a wide range of chemical characteristics, typically including asbestos, metals, and organic substances. DT also generally results in the generation of secondary radioactive waste which must be managed.

The destructive analysis process for waste package characterisation is a multistep process that should be optimised carefully to allow for a reliable characterisation of the waste packages. The first and the most important step is the sampling (sample preparation and chemical separation, radiological and chemical measurements), where representative samples should be collected to ensure the reliability of the characterisation results. Sampling needs to account for potential inhomogeneity of waste streams and therefore the sampling procedures in destructive analyses should carefully follow sampling plans established in advance. Subsequently, designing the sampling procedures and checking the homogeneity and representation of the samples is very crucial to ensure the success of the characterisation process, minimise the waste generation, and reduce the characterisation time [95].

Another important aspect is the representativeness of the samples, which is a key parameter for reliable waste characterisation. The accuracy and precision of analytical methods must be rigorously validated to ensure confidence in measurement results. However, the absence of matrix-matched certified reference materials for specific radionuclides significantly complicates the validation process. This scarcity can lead to increased measurement uncertainties, as laboratories lack standardised materials to benchmark their results [82].

The cost of measurements presents a substantial challenge that limits the number of analyses that can be performed. These high costs stem from multiple factors: the time-intensive nature of destructive testing (which can range from days to weeks per sample: the process involves multiple steps, including sampling, preparation, chemical separation, and measurement, each requiring significant time to ensure accuracy [20]), the requirement for highly specialised personnel with expertise in radiochemistry and nuclear instrumentation, and the need for expensive and complex equipment such as high-resolution ICP-MS, alpha spectrometers, and liquid scintillation counters [16].

Some destructive analytical methods are inherently complex or not fully optimised, which can affect their reliability. Challenges such as handling high-activity samples, ensuring chemical safety during sample digestion and separation processes, and achieving representative sampling require meticulous planning and method development [83]. The heterogeneity of nuclear waste matrices often necessitates extensive sample preparation procedures to minimise matrix effects and interferences, further increasing analytical complexity and cost.

## 5.1 Current Methods

In this section, some of the most relevant DTM radionuclides are reported. The selection has been driven by the lack of reliable analytical methods ( $^{79}\text{Se}$ ,  $^{93}\text{Zr}$ ,  $^{107}\text{Pd}$ ,  $^{243,244}\text{Cm}$ ), or by the availability of methods excessively time consuming ( $^{14}\text{C}$ ,  $^{36}\text{Cl}$ ,  $^{99}\text{Tc}$ ) or difficult to be standardised ( $^{41}\text{Ca}$ ,  $^{93}\text{Mo}$ ,  $^{135}\text{Cs}$ ), especially because certified standard materials are not commercially available. Moreover, these radionuclides are important for radioactive waste repository due to their long half-lives, high mobility in the repository site and environment as well as their relative high radioactivity after a long-time decay. The section provides the main characteristics and best available methods of these radionuclides. While the current analysis presents a targeted selection of critical radionuclides, it is acknowledged that list of

DTM radionuclides of concern for nuclear decommissioning and complete radiological characterisation of radioactive waste is longer and more variegated. However, for most of them, optimised and effective standardised procedures and commercial solutions are already available and are routinely used by radiochemical laboratories. Among others, we may enumerate  $^3\text{H}$ ,  $^{55}\text{Fe}$ ,  $^{59}\text{Ni}$ ,  $^{63}\text{Ni}$ ,  $^{90}\text{Sr}$ , uranium, thorium and plutonium isotopes within this category. These and other less challenging radionuclides have already been investigated and reported in previous projects. For these reasons, the reader is forwarded to previous literature [92], [109].

### 5.1.1 Determination of $^{14}\text{C}$

#### 5.1.1.1 Origin and characteristics

$^{14}\text{C}$  is a long-lived pure  $\beta$ -emitting radionuclide. It is a relevant radionuclide for the long-term safety of disposal facilities due to its relatively long half-life ( $T_{1/2} = 5730$  years). It is primarily produced by neutron activation of nitrogen through the reaction  $^{14}\text{N}(n,p)^{14}\text{C}$ , where  $^{14}\text{N}$  has a natural abundance of 99.6% and a thermal neutron cross-section of approximately 1.8 b. In some reactor designs, nitrogen is not merely an impurity in the coolant or structural materials but is intentionally used as part of the coolant system, as in some gas-cooled reactors, where nitrogen or nitrogen-based mixtures are employed as coolant, contributing to  $^{14}\text{C}$  production. In RBMK reactors nitrogen gas is used for purging and blanketing purposes. Additionally, in reactors with graphite moderators,  $^{14}\text{C}$  can also be produced from carbon via neutron capture by carbon isotope  $^{13}\text{C}$ .  $^{14}\text{C}$  is also generated as a fission product in low yields. In radioactive waste,  $^{14}\text{C}$  can exist in various chemical forms, including inorganic carbonates and organic compounds, complicating the extraction and quantification processes. It decays by beta emission with a relatively low maximum energy ( $E_{\text{max}} = 156$  keV), making it a DTM radionuclide for radiological assessments in waste management and environmental studies [96],[97].

#### 5.1.1.2 Available measurement techniques

$^{14}\text{C}$  is usually determined in radioactive waste by Liquid Scintillation Counting (LSC) and Accelerator Mass Spectrometry (AMS) after appropriate chemical separation from interfering species [98]. New methods for rapid determining  $^{14}\text{C}$  are also under development. LSC requires the chemical removal of interfering beta-emitting radionuclides from the sample matrix. Solid samples, such as carbonaceous waste, typically need extensive pre-treatment steps, including combustion to transfer carbon into a liquid (usually carbon dioxide absorbed in a scintillation-compatible medium). Due to the complexity of sample preparation, analysis times can take several hours. AMS is a highly sensitive and precise analytical technique used for detecting long-lived radionuclides, including  $^{14}\text{C}$ , in various sample types [99],[100]. Its ability to directly count individual  $^{14}\text{C}$  atoms relative to stable isotopes ( $^{12}\text{C}$  and  $^{13}\text{C}$ ) provides superior sensitivity compared to LSC. AMS operates by converting the carbon content of a sample into graphite or  $\text{CO}_2$  gas. The sample undergoes chemical pre-treatment to isolate the carbon component. AMS requires relatively small sample sizes (as low as milligrams of carbon), which is advantageous for handling limited amounts of highly radioactive samples. However, sample preparation is complex and time-consuming, involving intensive chemical purification and physical conversion steps.

Optimisation of decommissioning activities requires not much time-consuming characterisation methods. For this purpose, a few methods are under development for rapid determining  $^{14}\text{C}$  in irradiated graphite [101],[102],[103],[104],[105],[106]. They are based on combustion of a solid sample by the dedicated elemental analyser and subsequent measurement of a purified gaseous  $\text{CO}_2$  sample.  $^{14}\text{C}$  activity can be measured by semiconductor beta detector, thermal conductivity detector [104] or optic analyser [105]. In particular, the methods based on cavity ring-down spectroscopy (CRDS) [104],[106] use the detection of the  $^{14}\text{CO}_2$  molecule using its P(20) absorption line in the mid-infrared wavelength region at  $2209.109\text{ cm}^{-1}$  to assess  $^{14}\text{C}$  activity. The total  $\text{CO}_2$  concentration in the cavity is obtained by measuring a  $^{13}\text{CO}_2$  line at  $2209.77\text{ cm}^{-1}$  and using the same line fitting method as for the radiocarbon

spectra in standard CRDS [106] or measurement of a reference material is used in saturated-absorption CRDS [104].

### 5.1.2 Determination of $^{36}\text{Cl}$

#### 5.1.2.1 Origin and characteristics

$^{36}\text{Cl}$  originates by neutron activation through the following reactions:  $^{35}\text{Cl}(n, \gamma)^{36}\text{Cl}$ , where  $^{35}\text{Cl}$  is the most abundant chlorine isotope and thermal cross section is  $\sigma = 10$  mb for thermal neutrons, and  $^{39}\text{K}(n, \alpha)^{36}\text{Cl}$ , where  $^{39}\text{K}$  is the main potassium isotope and thermal cross section 2 b. For this reason, in the presence of intense neutron fields materials like concrete, steel and graphite will be activated leading to the generation of  $^{36}\text{Cl}$ . This isotope of chlorine is an almost pure beta emitter ( $E_{\text{max}}$  709 keV, 98%) and has a half-life of  $3.01 \times 10^5$  y; these two characteristics earned  $^{36}\text{Cl}$  the DTM radionuclide classification.

The measurement of  $^{36}\text{Cl}$  is of particular importance for the correct classification of radioactive waste during decommissioning activities: the volatility of chlorine and its mobility pose a challenge for the safe disposal of radioactive waste, hence making the measurement of  $^{36}\text{Cl}$  of extreme importance.

#### 5.1.2.2 Available techniques of measurement

$^{36}\text{Cl}$  can be either measured by Accelerator mass spectrometry (AMS) or Liquid Scintillation Counting (LSC). AMS systems are extremely costly and available in a few research sites; on the other hand, LSC requires to perform a thorough radiochemical separation of the analyte to separate it from possible interfering species. Even more so, when dealing with samples consisting of complex matrixes as concrete samples from decommissioning, the need for *ad hoc* methods of radiochemical separation arises.

The measurement of  $^{36}\text{Cl}$  in concrete has already been addressed in the literature, e.g. by Hou and Ashton [107],[108]. The first method consists of an alkali fusion, a selective precipitation and dissolution, a chromatographic separation to further purify the analyte which is then measured by LSC with scintillation cocktail. The method by Ashton et al. aimed at the measurement of  $^{36}\text{Cl}$  and  $^{129}\text{I}$ , and, to do so, it proposed a leaching of the sample with a sodium hydroxide solution and a selective oxidation of the analytes to gases, then trapped in NaOH solutions. Then, the two solutions containing the purified analytes are measured by LSC.

### 5.1.3 Determination of $^{41}\text{Ca}$

#### 5.1.3.1 Origin and characteristics

$^{41}\text{Ca}$  is a long-lived ( $T_{1/2}=1.03 \times 10^5$  y) radionuclide decaying by electron capture without any gamma emission, it emits only low energy X-rays (3.3 keV, 11.4%) and Auger electrons (3.0 keV, 77%), which are not easily detected by LSC.  $^{41}\text{Ca}$  appears in nuclear waste, especially in concrete used as biological shielding in reactor buildings, after neutron activation of  $^{40}\text{Ca}$  in the  $^{40}\text{Ca}(n, \gamma)^{41}\text{Ca}$  nuclear reaction (target abundance 97 %, cross section  $\sigma = 0.4$  b). The  $^{41}\text{Ca}$  level in nuclear waste is of interest because of its long half-life and relatively high mobility in the environment.

#### 5.1.3.2 Available measurement techniques

$^{41}\text{Ca}$  must be completely separated from the sample matrix and any interfering radionuclides before measurement. The chemical separation of calcium from the sample matrix and other interfering radionuclides is a crucial process for accurate calcium measurement. Calcium is separated by precipitation. The general principle involves the following steps. First, alkali and alkaline-earth metals are separated from transition metals, which precipitate as hydroxides at a lower pH. Next, alkaline-earth metals are separated as carbonates or phosphates from the alkali metals. Finally, calcium is selectively separated from other alkaline-earth metals by the precipitation of calcium hydroxide. Available measurement techniques with MDA/Detection limits include: LSC ( $10^{-1}$  Bq/g of  $^{41}\text{Ca}$  using 1 g of concrete), AMS ( $10^{-6}$  -  $10^{-8}$  Bq/g of  $^{41}\text{Ca}$ ), ICP-MS/MS (0.32 Bq/g (0.099 ng/g).  $\text{Fe}(\text{OH})_3$  precipitations



are performed to remove various contaminants such as actinides, lanthanides, Fe, Co, Ni etc. by scavenging [109],[110],[111],[112],[113]. Many contaminants can be removed by anion exchange chromatography. The use of TRU resin for further purification of Ca from actinides and lanthanides was also proposed.

#### 5.1.4 Determination of $^{79}\text{Se}$

##### 5.1.4.1 Origin and characteristics

$^{79}\text{Se}$  is the only isotope of selenium of relevance from a waste management perspective. It is a long lived, low energy pure beta emitter ( $T_{1/2} = 3.27 \times 10^5$  y,  $E_{\text{max}} = 150$  keV), decaying to the ground state of stable  $^{79}\text{Br}$ . It is produced by radiative neutron capture on stable  $^{78}\text{Se}$  (abundance 23.7%, thermal cross section 0.4 b), and as a fission product (yield from thermal fission of U-235 <0.05%). Owing to these two routes of production,  $^{79}\text{Se}$  can be expected to be mainly present in activated steel structures, where its stable precursor can be used as an alloying element, and in irradiated fuel, as well as in any waste deriving from reprocessing activities.

Its radiological importance is linked to the low sorption of selenate ions to clay minerals, and hence to their high mobility in the biosphere. Actual speciation of Se in irradiated fuel is still a topic of investigation [114]. Anyhow, reduced forms can be easily oxidised to high-valence anionic species, which can be relevant for the assessment of repository long term safety.

##### 5.1.4.2 Available measurement techniques

$^{79}\text{Se}$  can be measured both via radiometric (LSC) and non-radiometric methods (ICP-MS or AMS), with corresponding different requirements in terms of radiochemical manipulation. Both approaches have been reported in literature [115],[116],[117].

Radiometric determination of  $^{79}\text{Se}$  must take into account the very low specific activity of the analyte, and the likely simultaneous presence of several high-activity matrix-specific interferents, the most typical being  $^{60}\text{Co}$ ,  $^{55}\text{Fe}$ ,  $^{63}\text{Ni}$ ,  $^{93}\text{Mo}$  for steel alloys. More diverse interferants could be expected for other types of sample matrices. For determinations relying on mass spectrometry, the isobaric interference from stable  $^{79}\text{Br}$  is to be removed with high decontamination factor.

Typical radiochemical purifications take advantage of the anionic form of the selenate to separate it from most cationic interferents via anion exchange chromatography. Removal of halides can be accomplished via precipitation with silver nitrate. An additional chromatographic separation on an anion exchange medium can be employed to remove interference from  $^{79}\text{Br}$ .

#### 5.1.5 Determination of $^{93}\text{Zr}$

##### 5.1.5.1 Origin and characteristics

$^{93}\text{Zr}$  is a long-lived pure  $\beta^-$  emitting radionuclide. It is a relevant radionuclide for the long-term safety of disposal facilities for its very long half-life time ( $T_{1/2} = 1.5 \times 10^6$  years). It is produced both as fission products and by neutron activation of stable Zr, through the reaction  $^{92}\text{Zr}(n, \gamma)^{93}\text{Zr}$ , where  $^{92}\text{Zr}$  natural abundance is 17.1% and thermal cross section is  $\sigma = 0.26$  b. Zr is the main component of Zr alloys employed as fuel cladding in thermal reactors, but it is also present in non-negligible amounts in concrete and other materials.  $^{93}\text{Zr}$  is a DTM radionuclide, it has a relatively low energy beta spectrum:  $E_{\text{max}} = 59.5$  keV (73%) and 90.3 keV (27%).

##### 5.1.5.2 Available measurement techniques

$^{93}\text{Zr}$  is a DTM that can be measured by LSC and ICP-MS after separation from interfering species. To unlock LSC measure, it is necessary to remove both the matrix and the radionuclides emitting X-rays, beta and Auger electrons. ICP-MS is hampered by the presence of stable Zr ( $^{92}\text{Zr}$  and  $^{94}\text{Zr}$ ) and by isobaric interferences due to stable  $^{93}\text{Nb}$  and radioactive  $^{93}\text{Mo}$ , usually present in activated steel samples [118].

After sample matrix destruction, which depends on the sample itself, Zr is usually pre-concentrated by precipitation (e.g. as  $ZrO_2 \cdot nH_2O$  or as  $BaZrF_6$ ), then it is purified from interferences by solvent extraction (e.g. using TBP, TOPO, or HDEHP ligands), ion exchange or extraction chromatography (e.g. with commercial TRU, UTEVA, TEVA and Zr resins) [119]. The chemical yield could be assessed by using stable Zr as carrier or the short-lived and gamma emitter  $^{95}Zr$  as radioactive tracer [109].

## 5.1.6 Determination of $^{93}Mo$

### 5.1.6.1 Origin and characteristics

$^{93}Mo$  is a long-lived radionuclide ( $T_{1/2} = 4.0 \times 10^3$  y) which decays purely by electron capture (EC) on  $^{93m}Nb$  (88%) and  $^{93}Nb$  (12%). During decay, Nb characteristic X-rays ( $E = 16.615$  keV, 41%, the most probable one), Auger and conversion electrons are emitted without gamma-rays of significant intensities. Hence,  $^{93}Mo$  is considered a DTM radionuclide.  $^{93}Mo$  is an artificial radionuclide produced mainly by the radiative capture reaction (thermal cross section is 0.45 mb) on  $^{92}Mo$ , a stable nuclide with natural abundance of 14.6%. Its production in nuclear reactors occurs in structural materials receiving high neutron fluence, since molybdenum is present as an alloying element in stainless steel like AISI type 316, in mass fraction of 2% to 3%.

The transition element molybdenum can exist in oxidation states ranging from 0 to +VI, with the most common being +VI and +IV. It is present with oxidation state +VI in slightly acidic to alkaline solutions, as molybdate ion  $MoO_4^{2-}$ , whereas it is present as polymolybdate ion in acidic solutions. Chemistry of molybdenum is complex and still not thoroughly understood.

### 5.1.6.2 Determination methods

Measurement techniques used for  $^{93}Mo$  quantification are: x-ray spectrometry, LSC and ICP-MS. Prior to the actual quantification, radiochemical separation from interfering species is mandatory. For both x-ray spectrometry and LSC techniques,  $^{93m}Nb$  is the main interfering nuclide, due to the same x-rays and Auger electrons emitted as in  $^{93}Mo$  EC decay. Another significant interfering nuclide is  $^{93}Zr$ , since it decays through  $\beta^-$  on  $^{93m}Nb$ . For ICP-MS quantification the two major isobaric interferences are  $^{93}Nb$  and  $^{93}Zr$  [120]. TEVA-resin is commonly used for isolating Mo from interfering species [121]. Other methods exploit anion-exchange resins, tributyl phosphate (TBP) extraction, selective precipitation with  $\alpha$ -benzoin oxime or alumina-column [122].

## 5.1.7 Determination of $^{99}Tc$

### 5.1.7.1 Origin and characteristics

$^{99}Tc$  is a long-lived pure  $\beta^-$  emitting radionuclide ( $T_{1/2} = 2.1 \times 10^5$  y). Its high half-life and mobility make it one of the most important radionuclides in long-term disposal of radioactive waste.  $^{99}Tc$  can be produced by thermal neutron-induced fission of  $^{235}U$  with a fission yield of 6.06%, making it relatively abundant among fission products. Another way to produce this radionuclide is through neutron activation of  $^{98}Mo$  to  $^{99}Mo$  ( $T_{1/2} = 66$  h), which decays  $\beta^-$  to  $^{99}Tc$ .  $^{99}Tc$  cannot be measured without radiochemical separation and its  $E_{max}$  is 294 keV. These are the main reasons why  $^{99}Tc$  is a DTM radionuclide.

### 5.1.7.2 Available measurement techniques

Both radiometric (mainly GM gas flow counter and LSC) and mass spectrometric (ICP-MS, TIMS, RIMS, AMS) techniques have been used for the measurement of  $^{99}Tc$  [123],[124]. The radiometric methods have a lower cost, higher reliability and easier operation compared to the mass spectrometry ones. However, they have a much longer counting time and higher detection limits due to higher background levels.

### 5.1.8 Determination of $^{107}\text{Pd}$

#### 5.1.8.1 Origin and characteristics

$^{107}\text{Pd}$  is a long-lived radionuclide ( $T_{1/2} = 6.5 \times 10^6$  y) which decays purely by  $\beta$ -decay on  $^{107}\text{Ag}$  ( $E_{\max} = 34$  keV) while no gamma-rays are emitted. For these reasons,  $^{107}\text{Pd}$  is considered a DTM radionuclide. It is an artificial radionuclide, in particular a fission product of both  $^{235}\text{U}$  and  $^{239}\text{Pu}$  (cumulative fission yields of 0.15% and 3.2%, respectively). It is a concern in radioactive waste management and long-term disposal due to its presence in spent nuclear fuel and, consequently, in the HLW coming from reprocessing.

#### 5.1.8.2 Available measurement techniques

$^{107}\text{Pd}$  can be quantified either by LSC or ICP-MS, after separation from interfering species. Main interfering species in ICP-MS determination are isobaric and polyatomic species, especially Ag isotopes, Zr and Y oxides, and Pd hydrides [124]. Pd is commonly purified by using dimethylglyoxime (DMG), usually exploited for selectively extracting or precipitating it [125]. Other methods exploit commercial Ni-resins (which contains DMG) for selectively separating Pd from LSC interfering radionuclides such as  $^{55}\text{Fe}$  and  $^{63}\text{Ni}$  [126].

### 5.1.9 Determination of $^{135}\text{Cs}$

#### 5.1.9.1 Origin and characteristics

Radiocaesium contamination comes from anthropogenic activities.  $^{135}\text{Cs}$  is a long-lived and low-abundant pure beta emitter with  $E_{\max} = 268$  keV. Because of its high yield fission product (6.9% from fission of  $^{235}\text{U}$ ), long half-life ( $T_{1/2} = 2.3 \times 10^6$  y), it is mainly found in spent ion exchange resins used for purification of the primary circuit. Due to the high mobility to biosphere,  $^{135}\text{Cs}$  is one of the major radionuclides responsible for the long-term environmental impact of a waste repository, thereby calling for accurate waste characterisation and environmental monitoring.

#### 5.1.9.2 Available measurement techniques

The radiometric methods (e.g. LSC and NAA) are not sensitive due to the presence of isotopic interferences with higher energy, especially  $^{137}\text{Cs}$ . The non-radiometric methods are more suited for  $^{135}\text{Cs}$  detection for the higher sensitivity of mass spectrometry, even though preliminary purification by radiochemical methods is paramount. Common isobaric or polyatomic interferences are  $^{135}\text{Ba}$  and  $^{137}\text{Ba}$ ,  $^{95}\text{Mo}^{40}\text{Ar}$ ,  $^{97}\text{Mo}^{40}\text{Ar}$ . Thermal Ionisation mass spectrometry (TIMS) and Accelerator Mass Spectrometry (AMS) are poorly affected by interferences but suffer of difficult accessibility and are too slow for routine analysis. Inductively Coupled Plasma mass spectrometry (ICP-MS) is versatile and popular, but it does not allow an effective suppression of interferences. QQQ-ICP-MS equipped with a collision cell is more suitable as it allows further purification of the analyte, leading to higher decontamination factors.

Currently, some laborious analytical methods based on the selective ammonium molybdophosphate polyacrylonitrile (AMP-PAN) ion exchanger combined with anionic and cationic resins are used to pre-concentrate caesium and purify it from isobaric (Ba) and polyatomic interferences (Mo and others). One of these methods was implemented by Zhu et al. to determine  $^{135}\text{Cs}$  in spent radioactive ion exchange resins. After performing leaching, separation and purification steps, a caesium recovery higher than 85% and a decontamination factor for Ba of  $10^6$  were achieved. This analytical method combined with a tandem ICP-MS/MS instrument allowed to determine concentrations of  $^{135}\text{Cs}$  and  $^{135}\text{Cs}/^{137}\text{Cs}$  ratios, thus enhancing suppression of interferences and improving detection limits ( $1.3 \mu\text{Bq/L}$  from 0.2 g of resin) [128]. An alternative solution to the current use of the AMP-PAN resin was recently found to avoid the release of large amounts of Mo interference in the stripped Cs, or the use of energy intensive processes to destroy concentrated ammonium salt present in the AMP-PAN stripping solutions. This new analytical method is based on the combined effect of a co-precipitation step by calcium phosphate along with a chromatographic separation on a Sr-resin to preliminarily remove matrix contaminants (including the

isobaric  $^{135}\text{Ba}$ ). Afterwards, Cs pre-concentration and further purification stages are carried out by a cationic resin. A Cs recovery > 70% and a  $\text{DF}_{\text{Ba}}$  up to  $10^8$  have been obtained, without any concern of potential Mo release, that generally calls for the employment of anionic resins [129]. This radiochemical method could be combined with an ICP-MS instrument equipped with a Collision Cell to enhance the overall Ba decontamination (> $10^{11}$ ) before determining  $^{135}\text{Cs}$  and  $^{135}\text{Cs}/^{137}\text{Cs}$  ratios.

#### 5.1.10 Determination of $^{243/244}\text{Cm}$

##### 5.1.10.1 Origin and characteristics

Am and Cm are radionuclides belonging to the minor actinides category. They are produced by some consecutive activation reactions, mainly on  $^{238}\text{U}$ , followed by  $\beta^-$  decays. All minor actinides are  $\alpha$  emitters and can be measured by  $\alpha$  spectrometry. From the radiological point of view and for the higher production rate,  $^{241}\text{Am}$  is the most important minor actinide. It can be measured by alpha spectrometry, gamma spectrometry (with lower sensitivity), and mass spectrometry [130]. A more difficult task is the determination of  $^{243}\text{Cm}$  and  $^{244}\text{Cm}$ . Their half-lives are 29 and 18 years, respectively. Their main  $\alpha$  energies are 5785 keV ( $^{243}\text{Cm}$ ) and 5805 keV ( $^{244}\text{Cm}$ ), very difficult to be resolved by  $\alpha$  spectrometry. Even if the activity of  $^{243}\text{Cm}$  is usually neglected for its lower production rate, accurate estimation would be necessary.

##### 5.1.10.2 Available measurement techniques

Direct measurement of  $^{244}\text{Cm}$  via neutron detection in radioactive waste packages coupled with SF approach would simplify and improve the quantification of alpha activity in nuclear power plant waste packages. This approach would eliminate the need for sampling to evaluate alpha activity and would enable quick full-package assessment, thereby addressing issues such as sample representativeness and related challenges. Indeed, continuous analysis of waste samples has demonstrated that the correlation between TRUs from the same nuclear power plant is very strong and independent of the waste stream. For this reason, once the correlation between TRUs in a nuclear power plant is established, the direct measurement of  $^{244}\text{Cm}$  using NDT (neutron detection) would enable the determination of the activity of other TRUs, thus allowing the quantification of the alpha activity of the waste package. The main drawback of this method is that in radiochemistry,  $^{243}\text{Cm}$  and  $^{244}\text{Cm}$  cannot be easily measured separately. Therefore, an additional step would be required to determine the specific relationship between these isotopes for each nuclear power plant. This could be achieved using combined techniques.

A promising approach to measure  $^{243}\text{Cm}$  is to combine alpha and mass spectrometry. The presence of the isobaric interference  $^{243}\text{Am}$ , which occurs together with  $^{243}\text{Cm}$  and is difficult to remove due to similar chemical behaviour, hinders the determination of  $^{243}\text{Cm}$  by mass spectrometry, while  $^{244}\text{Cm}$  can be measured. The  $^{244}\text{Cm}$  activity obtained by mass spectrometry can be subtracted from the summed  $^{243}\text{Cm}+^{244}\text{Cm}$  activities obtained by alpha spectrometry to obtain a reliable estimation of the  $^{243}\text{Cm}$  activity in the sample. Among mass spectrometry techniques, AMS is the most sensitive one since the measure is not influenced by the presence of molecular isobars and matrix effects are not severe.

## 5.2 Case study

### 5.2.1 Ukrainian Case Study

#### 5.2.1.1 Current Challenges

- Significant volumes of accumulated historical waste require characterisation:
  - Approximately 42,000 m<sup>3</sup> at operating NPPs
  - Approximately 2,500 m<sup>3</sup> at the Chernobyl NPP

**EURAD-2** Deliverable 5.1 – State-of-the-art on innovative non-destructive techniques, destructive techniques, scaling factors for use cases

- Previous classification protocols did not require mandatory determination of nuclide composition (both gamma and DTM)
- Waste was primarily sorted based on dose rate measurements and stored in bulk, complicating characterisation
- Chernobyl NPP waste characterisation is further complicated by possible presence of emergency waste from the 1986 accident

#### 5.2.1.2 Future Development Opportunities

- Need for improved laboratory analysis methods to determine DTM
- Potential application of ICARUS WP5 study outcomes for proper characterisation of emergency RW
- Opportunity for optimisation of characterisation process by combining NDT methods with SF approaches
- Requirements for preliminary measurements during waste retrieval:
  - Gamma-emitting nuclides detection
  - Total alpha and beta activity measurement
  - Volume consideration factors

#### 5.2.2 Dutch Case Study

As summarised in the IAEA Country Nuclear Power Profiles 2019 edition, that the Netherlands has one nuclear power reactor in operation, one nuclear power plant in safe enclosure, two research reactors, one enrichment plant (URENCO) and one central storage facility for radioactive waste (COVRA) [131].

##### 5.2.2.1 Current Reference Projects

- Historical waste management at NRG PALLAS site in Petten:
  - Mixed waste (stored since 1961) with no alpha emitting waste has been successfully retrieved, opened, sorted and characterised
  - Combined approach using NDT, laboratory analysis, and SF methods
    - LLW fraction transported to COVRA facility
    - ILW fraction repackaged and stored at NRG PALLAS for future transport [134]

##### 5.2.2.2 Future Development Plans

- Upcoming project (2025) for waste suspected to contain traces of fissile-related DTM-nuclides
- Implementation of specialised alpha-waste-hot-cell techniques:
  - Inner glove box for alpha-emitting nuclide containment
  - 10 cm lead outer shielding for gamma radiation management
- Reduction in processing rate from previous champagne to check for fissile related objects (4x35 litre drums per week in 2025 vs one per day in 2014) [132].



## 6. SF Methods

The SF method is an evaluation technique to determine the activity concentrations of DTM nuclides (those emitting low energy photons, pure beta emitters or alpha emitting nuclides) based on the correlation between them and the easily measurable gamma emitters nuclides.

This method can be applied for burial disposal of LILW, when the radioactivity of specific nuclides in waste packages must be declared in accordance with limits and criteria derived from safety assessment of the disposal facility.

The objective of this section is to summarise the key points regarding the application of SF for waste characterisation. Through the analysis of various international reference regulations, guides and European projects, it is sought to identify current trends, best practices and existing challenges in this field.

The objective of this section is to provide a global vision of the methodologies used internationally, the progress made, and the areas that require further research.

### 6.1 Overview of major SF methodology guidelines

In this section, an introduction to the content of the most relevant international projects and references regarding the use of SF is provided, describing the purpose and application and including in a schematic way the methodology that is developed in each case. Three key sources provide the foundation for SF methodology implementation in radioactive waste characterisation: ISO 21238:2007 [13], IAEA TECDOC NW-T-1.18 [17], and the EU PREDIS project [135]. These frameworks, while aligned in fundamental principles, offer complementary perspectives and varying levels of detail on implementation strategies.

All three methodological frameworks recognise the SF method as essential for determining DTM radionuclide activities in waste packages. However, they address different aspects of the methodology with varying emphasis:

- ISO 21238:2007 [13] provides guidelines for determining the radioactivity of DTM radionuclides by correlating them with easily measurable radionuclides (key nuclides). This standard primarily applies to waste from water-cooled reactor nuclear power plants but may extend to other reactor types. It presents a detailed framework for applying the SF method to estimate the radioactivity of DTM nuclides in low- and intermediate-level radioactive waste at nuclear power plants. This international standard presents guidelines on the empirical SF method and provides a basic flow of application for the SF method, highlighting the importance of representative and homogenised samples for reliable results and describing two evaluation methodologies for SF determination: by linear relationship and by non-linear relationship. Annex A of the document provides cross-regional case studies illustrating the method's application, showing how reactor design, operational history, and waste composition affect assessment accuracy. This structured approach ensures compliance with safety regulations and supports robust radioactive waste management practices.
- The IAEA Nuclear Energy Series No. NW-T-1.18 [17] aims to develop a standardised method to estimate the activity of radionuclides in waste where direct measurement is impractical. It focuses on DTM radionuclides, such as alpha and beta emitters, by correlating them with easy to measure (ETM) radionuclides, typically gamma emitters like  $^{60}\text{Co}$  and  $^{137}\text{Cs}$ . While primarily intended for LILW in nuclear power plants, this methodology is also applicable to research reactors, fuel processing facilities, decommissioning waste, and historical waste. The report provides comprehensive information on international experience in the determination and use of SF, following the ISO 21238:2007 [13] guidelines. It presents valuable international experience with the SF methodology that can be applied to evaluate the radioactive inventory of DTM nuclides in waste packages across various nuclear facilities. Key steps in the process include sampling, radiochemical analysis, and verification of correlations. The IAEA emphasises

the importance of methodological rigor to avoid overly conservative estimates that could prematurely limit repository capacity. The report highlights that collaborative data sharing between facilities and robust quality control throughout the process are essential to improve accuracy and manage uncertainties in radioactive waste characterisation.

- The PREDIS (Pre-Disposal Management of Radioactive Waste) project [135] was a European research initiative focused on the management of radioactive waste. This project was developed within the framework of the HORIZON 2020 Program of the European Union, from 2020 to 2024. The main purpose of PREDIS was to improve strategies and technologies for the management of radioactive waste before its final disposal. The project focused particularly on the phase prior to the final disposal of radioactive waste, covering aspects such as characterisation, treatment and minimisation of the waste. PREDIS Project [135] takes a broader technological innovation approach, placing SF methodology within the larger context of pre-disposal waste management. While not focused exclusively on scaling factors, it integrates SF methodology with other characterisation, treatment, and minimisation strategies.

## 6.2 Methodological approaches and implementation strategies

**Step 1 Preliminary Evaluation:** Plant characteristics (reactor type, reactor component materials, fuel performance history, mechanism through which nuclides are produced, variations in waste treatment and plant operational condition) and other factors, such as waste streams that affect the composition ratios between DTM nuclides and key nuclides are studied and SF classifications based on SF variability are assessed. Development of a representative sampling plan.

**Step 2 Sampling and Data Collection:** Appropriate sampling is carried out in accordance with the studies shown in STEP 1.

**Step 3 Correlation Analysis:** Using the nuclide analysis data, the correlation between DTM nuclides and key nuclides is observed through the use of scatter diagrams. SF groupings are studied considering influencing factors examined in STEP 1.

The applicability of the SF method for a particular grouping is determined based on whether there is an observable correlation. The samples of selected wastes are collected, and nuclide analyses are performed on these samples to establish the correlations. If there is no visually apparent correlation in the scatter plots, the data should be segregated by stream and examined in greater detail. It may be necessary in this case to calculate a representative mean value for the SF for each stream.

**Step 4 Activity Estimation:** The activity concentrations or total activity of key nuclides in each waste package to be assayed are determined by measuring the surface dose rate of the waste package and calculating a key nuclide activity using "dose-rate-to-activity" conversion calculation, or by gamma spectrometry or other means.

The activity concentrations of DTM nuclides are calculated based on the specific SFs and the appropriate key nuclides activity for each package.



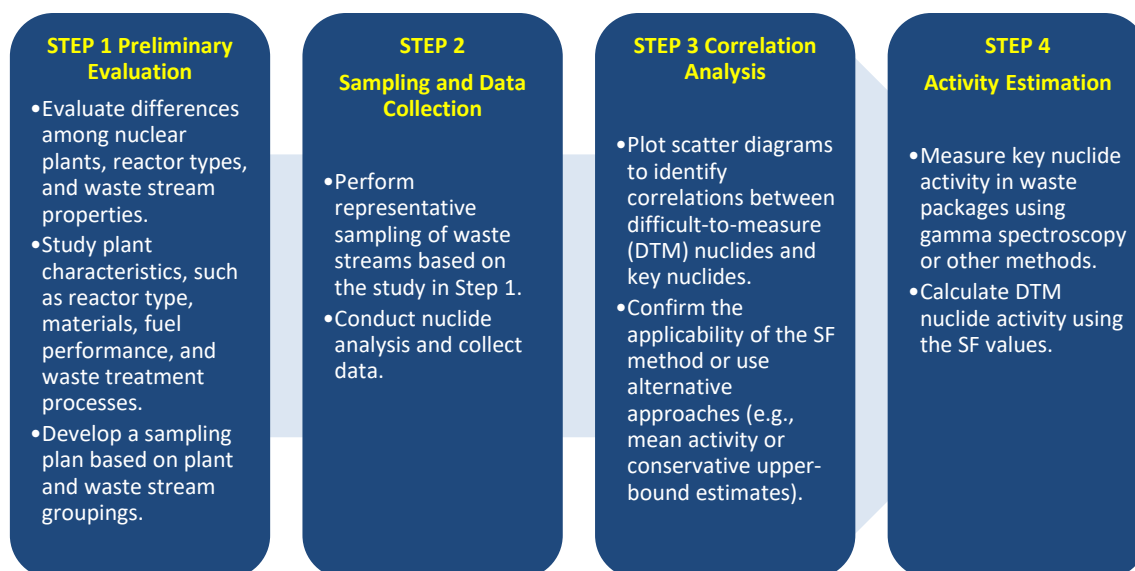


Figure 1 – SF Methodology according to ISO 21238:2007

As a summary of the case studies analysed from United States, European and Japanese waste:

- Key nuclide selection: in all cases, correlation between <sup>60</sup>Co and <sup>137</sup>Cs and fission-product nuclides and alpha-emitting nuclides were demonstrated for all plant types (PWR and BWR).
- Grouping by nuclear power plant type: The plant differences (reactor type, reactor component materials and fuel stability history) cause SF variations, so that may be considered in developing of the initial grouping.
- Grouping by waste stream: nuclide composition ratios of corrosion-product nuclides are relatively constant across various waste streams. Therefore, it can be possible to develop a unified SF for an entire plant. In case of fission-product nuclides, it is appropriate to consider the influence of the solubility of nuclides.

### 6.3 Comparative analysis of SF approaches in radioactive waste characterisation:

This document provides a systematic comparison of three primary methodologies for determining and applying SF in radioactive waste characterisation: ISO 21238:2007 [13], IAEA TECDOC NW-T-1.18 [17], and the PREDIS [135]. The tables below outline key aspects of these methodologies including theoretical and empirical approaches, sampling techniques, uncertainty management, and package-level applications

#### 6.3.1 Methodological framework comparison

Table 3 – Methodological Framework Comparison of Radioactive Waste Characterisation Standards

Aspect	ISO 21238:2007	IAEA TECDOC	EU Project PREDIS
Purpose	Evaluate correlations between key nuclides and DTM nuclides	Develop standardised method to estimate DTM radionuclide activity	Improve pre-disposal management of radioactive waste
Application scope	Waste streams with consistent correlations	LILW in nuclear power plants, research reactors, fuel processing facilities	Pre-disposal phase: characterisation, treatment, waste minimisation

Core principles	Statistical calculations of radionuclide correlations	Correlation of DTM with ETM nuclides following ISO guidelines	Collaborative approach between research institutions and industry
Time period	Originally published 2007	Based on ISO framework with expanded applications	European initiative 2020-2024

### 6.3.2 Theoretical vs. empirical approaches

Table 4 – Comparative Analysis of Theoretical and Empirical Approaches to SF Determination

Approach Type	Description	Advantages	Limitations	Key Considerations
<b>Theoretical</b>	Mathematical and computational modelling: nuclear reaction, activation calculations, contamination modelling	<ul style="list-style-type: none"> <li>- Useful when sampling data is limited</li> <li>- Can provide initial estimates</li> <li>- May consider physical processes systematically</li> </ul>	<ul style="list-style-type: none"> <li>- Often conservative</li> <li>- May not reflect actual conditions</li> <li>- Requires validation</li> </ul>	<ul style="list-style-type: none"> <li>- Must be validated with measurements</li> <li>- Helps adjust models to real-world conditions</li> </ul>
Statistical	Direct measurements of radionuclide activity in representative samples	<ul style="list-style-type: none"> <li>- Based on actual measurements</li> <li>- Captures real-world variability</li> <li>- More accurate when properly sampled</li> </ul>	<ul style="list-style-type: none"> <li>- Requires sufficient sampling</li> <li>- Sample representativeness critical</li> <li>- Can be resource-intensive</li> </ul>	<ul style="list-style-type: none"> <li>- Methods include geometric mean, regression analysis, logarithmic analysis</li> <li>- Robustness depends on sample size and representativeness</li> </ul>
Hybrid	Combination of theoretical modelling with empirical validation	<ul style="list-style-type: none"> <li>- Balances strengths of both approaches</li> <li>- Theoretical basis with real-world adjustment</li> <li>- Potentially most reliable</li> </ul>	<ul style="list-style-type: none"> <li>- More complex to implement</li> <li>- Requires both modelling and sampling</li> </ul>	<ul style="list-style-type: none"> <li>- Recommended by all three methodologies</li> <li>- Initial framework from models, refined by empirical data</li> </ul>

### 6.3.3 Statistical Methods for SF Determination

Table 5 – Statistical Methodologies and Techniques for SF Calculation Across Standards

Method aspect	ISO 21238:2007	IAEA TECDOC	PREDIS
<b>Primary methods</b>	<ul style="list-style-type: none"> <li>- Geometric mean (linear relationships)</li> <li>- Logarithmic regression (non-linear relationships)</li> </ul>	<ul style="list-style-type: none"> <li>- Log-log scatter plots</li> <li>- Geometric mean</li> <li>- Regression analysis</li> </ul>	<ul style="list-style-type: none"> <li>- Statistical means</li> <li>- Regression analysis</li> <li>- Advanced statistical tests</li> <li>- Bayesian framework</li> </ul>

Method aspect	ISO 21238:2007	IAEA TECDOC	PREDIS
Correlation approach	Observed correlations between key and DTM nuclides	Evaluation of DTM and ETM relationships using visualisation techniques	Adjusted for dispersion and variance of activity ratios
Data requirements	Sufficient data points with statistical verification	Coverage of significant waste streams and activity ranges	Statistical criteria for required sample numbers [150][151]
Quality control	Outlier rejection protocol using statistical methods	Regular updates based on new operational data	Higher correlation coefficients require smaller data sets
Statistical innovations	Statistical calculations to verify consistency	Integration with measurement techniques and plant-specific factors	- Fisher's test, binomial test, chi-squared test [152] - Bayesian updates with new data collection [19]
Special applications	Clear methodology for different correlation types	Adaptable to different reactor types	Interim SFs until finalised with complete data
Predictive capability	Standard approach for defined correlations	Better predictive behavior outside data range	Continuous improvement capability through updates

### 6.3.4 Uncertainty Sources and Management

Table 6 – Uncertainty Source Identification and Management Strategies in SF Methodologies

Uncertainty source	ISO 21238:2007	IAEA TECDOC	PREDIS
<b>Nuclide behavior</b>	Variations in behavior identified as key source	Variability from fuel failures, coolant chemistry	Considered in statistical framework
Measurement errors	Radiological analysis errors	Gamma spectrum or radiation level determination	Addressed through statistical techniques
Sampling variability	Addressed through statistical methods	Accounted for in correlation analysis	Minimised through optimised sampling
Package characteristics	Steps to assign package uncertainty from sampling uncertainty	Waste density, homogeneity, shielding differences	Similarity between package content and sampling space
Evaluation methods	- Number of data points - Standard deviation - Confidence intervals	- Geometric means - Log-normal distributions - Regular SF updates	- Correlation analysis - Variance calculations - Advanced statistical tests

Uncertainty source	ISO 21238:2007	IAEA TECDOC	PREDIS
Mitigation approaches	- Homogenised sampling - Conservative approaches for limited data	- Regular updates based on new data - Conservative values for high uncertainty	- Sufficient sample numbers - Bayesian framework for updates

### 6.3.5 Package-Level Application

Table 7 – Package-Level Application and Implementation of SF

Application Aspect	ISO 21238:2007	IAEA TECDOC	PREDIS
Measurement integration	Standard statistical methods for uncertainty propagation	Gamma spectrometry combined with SFs	Consideration of package-specific parameters
Uncertainty sources	Combination of sampling and measurement uncertainty	Measurement-related and package-specific uncertainties	Sampling uncertainty propagates to SF, causing inaccurate DTM nuclide activity calculations
Conservative approaches	Upper confidence limits for high uncertainty	Conservative values for regulatory compliance	Conservative confidence intervals
Scale effects	Not explicitly detailed	Statistical averaging across multiple packages	Lower package uncertainty due to larger mass
Compliance methods	Conservative assumptions for regulatory limits	Bounded uncertainty using confidence intervals	Enhanced collaboration with regulatory bodies

### 6.3.6 Comparison of reactor and waste type considerations

Table 8 – Reactor-Specific and Waste Type Considerations in SF Application

Consideration	ISO 21238:2007	IAEA TECDOC	PREDIS
Reactor type impact	Recognition of differences between PWR and BWR	Detailed consideration of reactor-specific factors	Part of broader waste characterisation framework
Waste homogeneity	Differentiation between homogeneous waste (e.g., liquid concentrates) and heterogeneous waste (e.g., mixed solid debris)	Waste groupings based on similarity	Specific methods for different waste types

Consideration	ISO 21238:2007	IAEA TECDOC	PREDIS
Operational history	Considered in record-keeping requirements	Significant factor in sampling plans	Considered in characterisation process
Waste treatment effects	Not explicitly detailed	Influence on radionuclide distribution noted	Focus area for waste minimisation

### 6.3.7 International Practice Examples

Table 9 – International Implementation Practices and Country-Specific SF Applications

Country	Key Focus Areas	Notable Practices	Referenced In
United States	Advanced implementation for operational reactors and decommissioning	Integration of empirical and theoretical models	IAEA TECDOC [17]
Japan	Reactor decommissioning	Extensive use of log-log regression for correlation refinement	IAEA TECDOC [17], [150], [151]
France	LILW disposal programs	Interim SFs until finalised with extensive analysis	IAEA TECDOC [17], [152]
Spain	Practical applications	LILW disposal program implementation	IAEA TECDOC [17]
Germany	Optimisation of sample size	Interim SFs for inhomogeneous waste	[152]
European collaboration	Pre-disposal management innovation	PREDIS project involving multiple countries	PREDIS [135]

### 6.3.8 Key Recommendations and Best Practices

Table 10 – Recommended Best Practices for SF Implementation and Optimisation

Area	Recommendations	Source
Sampling and analysis	<ul style="list-style-type: none"> <li>- Capture full activity concentration ranges</li> <li>- Use appropriate statistical techniques</li> <li>- Maintain detailed documentation</li> </ul>	All three methodologies
Statistical approach	<ul style="list-style-type: none"> <li>- Use confidence intervals and geometric means</li> <li>- Apply advanced statistical tests where appropriate</li> <li>- Consider Bayesian updates for new data</li> </ul>	Emphasised in PREDIS[135], [19],[152]

Area	Recommendations	Source
Methodology selection	<ul style="list-style-type: none"> <li>- Combine theoretical and empirical approaches</li> <li>- Customise to reactor type and waste stream</li> <li>- Validate all theoretical models</li> </ul>	All three methodologies
Uncertainty management	<ul style="list-style-type: none"> <li>- Propagate uncertainty systematically</li> <li>- Use conservative approaches for compliance</li> <li>- Consider scale effects between samples and packages</li> </ul>	All three, detailed approaches in IAEA TECDOC [17] and PREDIS [135]
International collaboration	<ul style="list-style-type: none"> <li>- Share data and best practices</li> <li>- Benefit smaller nuclear programs</li> <li>- Standardise approaches where possible</li> </ul>	IAEA TECDOC [17] and PREDIS [135]

## 6.4 SF Key Observations

Methodological Convergence across the three frameworks reveals an evolution in approach– from ISO's foundational methodology to IAEA's expanded applications and finally to PREDIS's collaborative innovation model, while maintaining shared core principles. The Sampling Focus shows distinct emphases, with IAEA highlighting customisation to plant-specific operational conditions and PREDIS introducing cost-efficiency considerations into sampling strategy development.

Uncertainty Treatment advances significantly with PREDIS, which establishes more precise statistical thresholds for determining sampling sufficiency compared to earlier frameworks. Similarly, PREDIS uniquely emphasises Regulatory Collaboration through proactive engagement with regulatory bodies to streamline compliance processes and uncertainty management.

The evolution toward Advanced Statistical Integration is evident in PREDIS's recommendation of specialised tests not explicitly covered in earlier frameworks, including Fisher's exact test, binomial test, and chi-squared test. PREDIS also introduces a Continuous Improvement Mechanism through its Bayesian update framework that enables systematic incorporation of new data, representing a significant advance in methodology sustainability.

Effective Operational Implementation requires comprehensive operator training and regular methodology updates based on emerging research and operational feedback. The Knowledge Transfer Focus highlights international collaboration as particularly vital for smaller nuclear programs with limited resources, underscoring the importance of accessible knowledge sharing platforms to support global best practices in radioactive waste characterisation.

## 7. Optimisation in industrial scenarios

Radiological characterisation represents a critical foundation for effective radioactive waste management across industrial applications. As regulatory requirements become increasingly stringent and disposal costs rise, optimising characterisation processes have emerged as a priority for both operational facilities and decommissioning projects. This section explores contemporary approaches to optimisation, focusing on methodologies that enhance accuracy while addressing operational constraints.

### 7.1 Decommissioning projects

Decommissioning of nuclear installations presents unique characterisation challenges due to complex radiological conditions, historical operations, and diverse waste streams. Optimisation strategies in this context focus on several key areas.

#### 7.1.1 Characterisation in decommissioning projects

Characterisation is a crucial stage in decommissioning projects as it ensures proper assessment, classification, and management of radioactive waste. It is an integral part of all decommissioning phases and should begin as early as possible. Effective implementation of this process requires clearly defined objectives and a structured approach.

The strategy and methodology of a characterisation program depend on the properties of radioactive waste (RW). Additionally, the accuracy and quality of the characterisation strategy are largely determined by requirements to demonstrate compliance with waste acceptance criteria for a specific disposal site, as well as acceptability criteria in some cases.

Identifying the life cycle of radioactive waste is a cornerstone in defining the strategy for RW characterisation. This is fundamental in shaping the approach to radioactive waste characterisation, as each stage – from generation to final disposal – requires defining and controlling key parameters.

All parties involved in the producing, processing, and disposal of RW, including the waste producer, waste processing operator, waste characterisation facility operator, repository operator, and regulator, should be involved in developing and detailing the characterisation strategy. The joint participation of these stakeholders ensures characterisation procedures comply with regulatory requirements, are implemented effectively, and have optimised costs [8].

#### 7.1.2 Implementation examples

The SF methodology is the most common technique for characterising solid RW as it's based on calculating the concentration of one radionuclide from known relationships with other, determined nuclides, which in turn simplifies RW characterisation at generation sites and thereafter. Defining and applying SF involves using various types of non-destructive and destructive radioactive waste analysis methods mentioned above.

Some general examples of implementing RW characterisation strategies for different types of nuclear facilities can be found in [91],[136]. For instance, in Germany's case, it is noted that decommissioning waste consists mainly of solid inorganic and organic materials and liquid inorganic substances that must be treated and conditioned properly. For this purpose, appropriate treatment facilities for combustion, compaction, evaporation, and drying must be available. This subsequently affects the processing methods and selection of RW processing facilities that need to be established for decommissioning.

Another interesting example is the Magnox reactors in the United Kingdom. It is noted that before dismantling this reactor, a complete characterisation needs to be performed.



### 7.1.3 Lessons learned

Regarding lessons related to organising the RW characterisation process for decommissioning purposes, the following key points can be highlighted [91],[136].

- Characterisation is fundamental for planning decommissioning activities and RW management
- No conditioning process should begin without prior detailed characterisation
- At the design stage, radiological and physicochemical characteristics of all possible waste streams should be considered in detail, including both primary and secondary waste
- During operations, an appropriate characterisation process for radioactive materials must also be ensured
- Integration of gamma spectrometry with advanced modelling techniques
- Development of portable and in-situ measurement systems
- Implementation of imaging technologies for radionuclide mapping
- Multivariate analysis of measurement data to resolve complex spectra

## 7.2 Operational Processes

To ensure operational control over the characterisation process and subsequently maintain an integrated RW management process through all stages, continuous monitoring of waste characteristics and quality assurance must be implemented.

Radioactive waste characterisation control can be provided through stationary automated control systems specified in the design, portable instruments, mobile installations, and laboratory testing. This control system must reliably determine radioactivity, chemical, physical, mechanical, thermal, and biological properties [8].

Quality assurance for the characterisation process is achieved by implementing a quality assurance programme for predisposal management [137], which should include measures for waste characterisation, confirmation of waste package characteristics, and review of quality control records. However, the primary responsibility for conducting quality waste characterisation rests with the waste producer [8].

For facilities managing ongoing waste generation, optimisation focuses on integrating characterisation into operational workflows through:

- **Process-integrated systems:** In-line monitoring systems, automated segregation technologies, digital twins incorporating characterisation data, and real-time decision support systems that minimise handling while maximising characterisation quality.
- **Knowledge-based systems:** Process Knowledge databases linking operational parameters to waste characteristics, Acceptable Knowledge frameworks for routine waste streams, expert systems, and machine learning applications that leverage existing information to reduce measurement requirements.
- **Quality Management optimisation:** Graded approaches based on waste classification, facility-specific uncertainty budgets, regular validation of SF, integration with facility-wide quality management systems, and continuous improvement processes that balance regulatory compliance with operational efficiency.

## 7.3 Case studies

### 7.3.1 Approaches in Decommissioning Projects

Decommissioning projects benefit from several optimisation strategies:

- Comprehensive preliminary characterisation mapping programs
- Statistical optimisation of sampling plans using Bayesian techniques
- Integration of multiple measurement technologies (gamma spectrometry, neutron coincidence counting, active neutron interrogation)
- Custom Monte Carlo modelling for complex geometries
- Historical data mining to establish radionuclide relationships

These approaches enable more precise waste categorisation, facilitate waste volume reduction, and support accelerated project timelines.

### 7.3.2 Approaches for operational waste management

Operational facilities can implement various optimisation techniques:

- Standardised measurement protocols based on waste stream characteristics [8]
- Development of facility-specific efficiency calibrations using computational methods [16]
- Integration of characterisation data with waste management databases
- Implementation of graded measurement approaches based on initial screening results
- Workflow optimisation to reduce handling and cross-contamination risks

These methodologies enhance throughput for routine waste packages while maintaining characterisation quality and regulatory compliance.

### 7.3.3 Approaches for special waste streams

Special waste streams, such as those from research facilities or non-standard operations, require tailored optimisation approaches:

- Development of facility-specific radionuclide vectors based on material composition and operational parameters
- Implementation of optimised multi-detector measurement systems
- Integration of analytical studies with direct measurements
- Development of custom algorithms for challenging radionuclide identification
- Correlation techniques for DTM

These specialised approaches enhance characterisation capabilities while addressing the unique challenges of non-standard waste streams.

### 7.3.4 Country specific case studies

#### 7.3.4.1 Ukrainian industrial optimisation

##### **Radioactive Waste Management Facilities of the SSP "Radon Association":**

- Similar bulk storage challenges with generally absent inventory information
- National strategy for waste retrieval requires optimisation approach
- Proposed in-situ characterisation workflow:

**EURAD-2** Deliverable 5.1 – State-of-the-art on innovative non-destructive techniques, destructive techniques, scaling factors for use cases

- Initial gamma screening to identify "hot spots"
- Application of various targeted characterisation methods
- Clearance-focused waste characterisation planned at operating NPPs and Chernobyl NPP
- Key optimisation challenge: determining optimal configuration of gamma scanners for waste characterisation equipment (for drums and containers of various geometries)

#### 7.3.4.2 Dutch Industrial Optimisation

- Construction of new Multifunctional Storage Facility (MOG) at COVRA (started 2024) ) [133].
- Facility designed for storage optimisation until 2050:
  - Accommodates expected waste streams over coming years
  - Storage of low-to-medium-activity waste canisters in stackable containers
  - Primarily intended for historical waste from Petten and future waste from all Dutch nuclear facilities
- Optimisation of methods for larger waste packages and high-rate processing:
  - Transportation and interim storage criteria compliance
  - Reuse and extension of proven LLW solutions for other waste streams
  - Scale-up considerations for processing volumes

## 7.4 Performance Data

Performance data in radioactive waste management is critical for ensuring efficiency, cost-effectiveness, and safety when implementing innovative characterisation techniques. Key performance indicators (KPIs) include efficiency metrics, cost analysis, and safety indicators, which are essential for assessing the viability, reliability, and economic feasibility of different techniques used in decommissioning projects and waste characterisation.

### 7.4.1 Efficiency metrics

Efficiency metrics in radioactive waste characterisation focus on improving detection sensitivity, data processing speed, and automation levels in both NDT and DT. Based on [91], the following efficiency metrics can be formulated:

- **Processing rate:** Measures the amount of radioactive waste processed per unit of time
- **Waste volume reduction:** Evaluates the effectiveness of treatment techniques in reducing waste volume before disposal
- **Characterisation accuracy:** Measures the success rate of identifying and categorising waste components accurately
- **Resource utilisation:** Tracks manpower and equipment use to optimise operations

A case study from the OECD NEA outlines a structured methodology for characterising radioactive waste that integrates statistical methods, historical records, and in-situ measurements to optimise efficiency. For example, the integration of automated systems in radioactive waste characterisation has led to significant reductions in both processing time and costs. According to [2], implementing automated characterisation technologies significantly reduce processing time compared to traditional manual methods.

#### 7.4.2 Cost considerations with regard to waste characterisation

Waste characterisation represents a significant investment within the overall radioactive waste management lifecycle that can influence downstream costs and efficiencies. While characterisation activities themselves constitute a relatively small portion of total waste management expenditures, their impact on the overall economic efficiency of the process is substantial.

Radioactive wastes should be characterised using the best available techniques so as to facilitate their subsequent management, including waste disposal [138]. High-quality characterisation can reduce uncertainties within the RW management cycle and decrease processing and disposal costs.

Comprehensive and accurate waste characterisation provides several economic benefits across the waste management lifecycle:

**Optimised waste classification:** precise characterisation prevents conservative over-classification of waste, reducing disposal costs for materials that can be managed at lower-tier disposal facilities or potentially cleared

**Reduction in processing uncertainties:** well-characterised waste streams allow for more efficient treatment processes and reduced conservatism in stabilisation requirements

**Prevention of repackaging or rework:** accurate initial characterisation helps avoid costly repackaging or additional treatment steps if waste acceptance criteria are not initially met

**Enhanced disposal efficiency:** detailed knowledge of waste properties enables optimised packaging and more efficient use of disposal space

According to [8], strategic characterisation planning can result in 15-30% cost reductions in subsequent waste management operations.

From cost, efficiency, health and safety and environmental perspectives, it is recommended the characterisation approaches to acquire new information be considered in the following order of priority [138]:

- characterisation by calculation;
- characterisation by NDT; and
- characterisation by sampling and analysis.

While underfunding of characterisation activities may reduce immediate costs, the NEA's analysis of decommissioning projects indicates that inadequate characterisation frequently leads to significant cost escalations later in the waste management lifecycle [139]. Proper waste characterisation and categorisation are critical for successful decommissioning. Well-executed campaigns can reduce disposal volumes by up to tenfold, significantly lowering overall decommissioning and waste disposal costs.

It is important to note that contingency allocations in waste management programs are typically applied at the program level rather than specifically to characterisation activities. However, improved characterisation data quality directly contributes to reducing overall program contingency requirements by decreasing uncertainty.

Ultimately, the economic value of waste characterisation lies not in minimising characterisation costs, but in optimising the information obtained to enable cost-effective decisions throughout the remainder of the waste management lifecycle. This economic optimisation must be achieved while satisfying regulatory principles that mandate the use of best available techniques for characterisation, which provides a framework for balancing cost considerations with safety and environmental protection requirements [140].

### 7.4.3 Safety Indicators

Safety indicators are quantifiable parameters derived from characterisation data that enable the assessment of waste safety throughout its management lifecycle. These indicators serve as metrics for evaluating risk levels associated with storage, transportation, and final disposal, while demonstrating compliance with regulatory requirements and optimising waste management processes. They help evaluate risk levels for personnel, the environment, and the public, as well as the effectiveness of applied safety measures. According to the IAEA Safety Standards [140],[141], safety indicators provide measurable evidence for safety cases and are dependent on accurate waste characterisation.

Examples of information needed to ensure safety include:

#### **Radiological properties**

- Radionuclide-specific activities: identification and quantification of radionuclide inventories providing source term data for safety assessment models
- External dose rates: surface and volumetric measurements informing handling protocols, shielding requirements, and transport classifications

#### **Chemical and physical properties**

- Flammability indicators: assessment of hydrogen generation potential through radiolysis and reactive metal interactions
- Gas generation potential: determined through characterisation of organic content, moisture levels, and chemical composition to predict long-term behaviour
- Chemical compatibility: identification of substances that may react with packaging materials or other waste components
- Physical stability: determination of mechanical properties and long-term durability under storage and disposal conditions

#### **Process and Environmental Monitoring**

- Material flowsheet mapping: determination of radionuclide partitioning between solid waste products, liquid effluent, and gaseous effluent
- Environmental impact tracers: monitoring of where radionuclides and hazardous chemical substances ultimately reside
- Accumulation detection: identification of potential long-term radionuclide accumulations that may go undetected by routine process monitoring due to analytical precision limitations

Radioactive waste should be properly characterised and segregated to facilitate its subsequent safe and effective management within a quality framework using a systematic approach to acquire data sufficient for waste management decisions throughout the lifecycle.

These regulatory principles clearly establish characterisation as a fundamental requirement for safety case development.

For waste disposal or transfer, waste must comply with all radionuclide properties, physical properties, and chemical properties are essential to demonstrate compliance across this full spectrum of safety-critical parameters.

## 8. Technical gaps to address

Despite significant advances in radioactive waste characterisation over recent decades, several critical technical gaps remain that limit the efficiency, accuracy, and cost-effectiveness of current methodologies. This section identifies key technical limitations in both NDT approaches and physical-chemical characterisation methods.

### 8.1 NDT enhancement

#### 8.1.1 Detection sensitivity limitations

Current NDT methods face significant challenges in accurately detecting low levels of radionuclides, particularly in complex waste matrices. Gamma spectrometry struggles with very low energy gamma photons or electron capture decaying radionuclides such as  $^{55}\text{Fe}$ ,  $^{59}\text{Ni}$ ,  $^{93}\text{Mo}$ , that emit only soft X-rays that experience severe attenuation within the waste matrix. The attenuation problem, combined with self-shielding effects in dense materials, creates spatial detection biases where activity concentrations may be underestimated by factors of 2-10 depending on matrix composition and radionuclide distribution.

Technical detection issues further compromise measurement accuracy, spectral interference between similar-energy emissions, coincidence summing losses for complex decay schemes, and elevated background thresholds that mask low-activity components. The heterogeneous distribution of radionuclides within waste packages introduces additional uncertainty, as hotspots may be missed entirely or their contribution misrepresented depending on their spatial relationship to detection systems [8]. Research indicates that even advanced gamma scanning systems can have detection uncertainties exceeding 30% for heterogeneous waste packages with varying density distributions [142]. These sensitivity limitations often result in conservative overestimation of activity inventories and significant detection challenges when characterising radioactive waste drums using gamma scanning systems, particularly for low-activity waste with complex matrices [143].

#### 8.1.2 Processing speed constraints

The time required for comprehensive NDT characterisation represents a substantial bottleneck in waste management workflows. Conventional segmented gamma scanning (SGS) of a standard 200-litre waste drum typically requires 1-3 hours per package for adequate statistical confidence, making the characterisation of large waste volumes generated during decommissioning projects prohibitively time-consuming [8].

In Tomographic Gamma Scanning (TGS), signal processing difficulties arise due to the low count rate recorded at each energy line. This is particularly critical when measuring standard containers with radioactive waste, as obtaining a sufficiently accurate image requires more than 8 hours. The cause is the low activity of the  $^{152}\text{Eu}$  source (~2.29 MBq), which reduces the radiation signal level, especially for high-density materials such as cemented waste (CMT).

Measuring low-energy nuclides, such as  $^{241}\text{Am}$  (59.7 keV) and  $^{133}\text{Ba}$  (81 keV), is complicated by insufficient protection against background noise. The detector's protective shutter does not effectively screen background radiation, leading to distortions in the spectra. Additionally, using a tungsten collimator in a system with an HPGe detector creates interference in  $^{241}\text{Am}$  determination, as its main line (59.54 keV) overlaps with tungsten's characteristic X-ray lines (59.32 keV and 57.98 keV), reducing measurement accuracy [144].

Critical review of characterisation techniques implemented at CEA France, noted that radiological characterisation is in constant evolution because of the increasing demand in terms of precision and sensitivity, yet computational capabilities have not kept pace with these increasing demands [16].



### 8.1.3 Geometry handling deficiencies

Two main approaches are used when measuring nuclide activity in radioactive waste, determined by the detection geometry type and scanning method. The first approach is open detection geometry, applied for IGS. The second is collimated detection geometry, used for Segmented Gamma Scanning SGS.

Considering the packaging geometry or large-sized material, it's important to select the appropriate gamma detector movement mode during measurement. Various mechanisms can be applied for this purpose:

- Rotating the sample on a turntable to average radial and angular variations in the system for heterogeneous waste
- Linear movement of the detector (or sample).

Additionally, when using segmented scanning, it is necessary to correctly determine the size of the segment to be analysed, as segments that are too large can lead to loss of spatial resolution, while segments that are too small can increase measurement time without significantly improving result quality [8].

A major limitation of current NDT methodologies is their restricted applicability to standardised waste package geometries. Most operational systems are optimised for specific container types (typically 200-litre drums) and struggle to accurately characterise non-standard containers, large components, or irregularly shaped waste items.

The geometry gap is particularly evident in the characterisation of decommissioning waste, which often includes large structural components, complex equipment assemblies, and irregularly shaped debris. While recent innovations such as the portable geometry-independent tomographic system show promise, they remain at laboratory scale and have not yet been implemented in routine waste management operations, the state-of-the-art of NDT for in-situ radiological characterisation remains limited by geometry constraints, particularly for complex structures encountered during decommissioning [147].

### 8.1.4 Data analysis automation inadequacies

Current NDT methodologies still rely heavily on expert interpretation and manual intervention in data analysis workflows. Spectrum analysis for gamma spectrometry, particularly for complex mixed radionuclide fingerprints, typically requires specialist interpretation to resolve peak overlaps, account for interferences, and address matrix effects.

Gamma ray scanning can be performed in a highly automated way with only minimal operator interaction. Due to the complexity of the applied equipment and procedures, it must be accompanied by quality control and quality assurance protocols. This procedure must ensure correct accounting for gamma radiation attenuation effects caused by absorption in RW materials, the immobilisation matrix, container walls, and background radiation [8], [144].

According to the EURAD WP9 findings, less than 30% of European waste management facilities employ fully automated analysis systems for NDT data interpretation [145]. Machine learning, a subfield of AI, uniquely derives relationships and rules from data, enabling machines to tackle complex problems and manage uncertainty. This capability has driven its research applications in engineering as a fast estimation and optimisation tool. However, a significant gap exists in implementing AI and machine learning approaches for waste characterisation. While numerous learning-based methods have been proposed, understanding both their potential benefits and implementation challenges will help researchers better formulate problems and collect representative data for robust applications [146].



## 8.2 Physical-chemical characterisation

### 8.2.1 Limited real-time capabilities

Current methodologies for physical and chemical characterisation of radioactive waste predominantly rely on laboratory analysis of extracted samples, creating significant delays between sampling and results availability. According to [4], typical turnaround times for comprehensive chemical analysis range from several days to weeks, introducing operational delays and creating bottlenecks in waste processing workflows [12].

Larijani et al. observed that while radiological characterisation has seen significant advances in field-deployable technologies, "chemical treatments are tedious, time-consuming and require significant amounts of radioactive samples leading to exposure of operators to substantial doses and causes problems for waste management, such as contaminated organic solvents" [6].

### 8.2.2 Non-destructive methods limitations

Non-destructive methods for physical-chemical characterisation of radioactive waste remain significantly less developed than their radiological counterparts. Current approaches for identifying hazardous chemical constituents rely heavily on historical knowledge and waste stream provenance data rather than direct measurement.

Mauerhofer et al. (2023) pointed out a significant limitation in current non-destructive characterisation technologies (including gamma scanning, X-ray imaging, neutron counting methods, and muon tomography), noting their inability to detect and identify non-radioactive hazardous materials in waste packages, which represents a critical gap in waste characterisation capabilities [142].

### 8.2.3 Automation level deficiencies

Physical-chemical characterisation workflows remain predominantly manual and labour-intensive, with limited integration into automated waste processing systems. Very few of European waste management facilities employ robot-assisted sampling systems, despite their potential to reduce worker exposure and improve sampling representativeness.

Data integration between physical-chemical characterisation and radiological characterisation represents another critical automation gap. Most facilities operate these as separate analytical workflows with manual data transfer between systems, creating opportunities for transcription errors and preventing integrated data analysis.

Research in digitalisation for nuclear waste management in [148] highlighted that despite significant advances in data science, artificial intelligence, and automation in other industries, waste characterisation remains largely reliant on traditional, manual approaches with limited digital integration.

### 8.2.4 Cost efficiency challenges

The economic viability of comprehensive physical-chemical characterisation represents a significant obstacle to implementation, particularly for large waste volumes generated during decommissioning.

The IAEA's publication on "Methods for the Minimisation of Radioactive Waste from Decontamination and Decommissioning of Nuclear Facilities" emphasises that "characterisation costs can represent a significant portion of overall waste management budgets, creating economic pressure to minimise characterisation scope despite technical arguments for more comprehensive approaches" [149].

## 8.3 DTM Analysis

Several limitations have been highlighted for efficient and sustainable radiological characterisation of DTM radionuclides, e.g. time-intensive sample preparation, complex matrix and interference removal, unsatisfactory detection limits, and excessive analysis costs. Moreover, in some cases, the lack of

certified standard materials hinders the formal validation of the protocols, resulting in unacceptably high uncertainties and inaccuracy. The main limitations and open challenges are reported in the following paragraphs for the selected DTM radionuclides of interest.

An additional challenge is the lack of intercomparison exercises and of commercial reference materials to be used to validate, demonstrate and harmonise the developed analytical methods for determination of DTM radionuclides. The intercomparison would allow performance assessment of the novel methods and of the participating laboratories. In several cases, reference materials do not exist yet (e.g. for  $^{93}\text{Mo}$ ,  $^{93}\text{Zr}$ ,  $^{107}\text{Pd}$ ). Hence, alternative solutions should be provided, for example identifying real waste samples collected during NPP decommissioning (e.g., ion exchange resin, concrete, alloys, graphite) to be distributed to several radiochemical laboratories.

#### 8.3.1 $^{14}\text{C}$ main challenges

Determining  $^{14}\text{C}$  in radioactive waste presents several challenges due to its low energy emissions, several radiometric interferences, and the complexity of methods and waste matrices. Accurate determination of  $^{14}\text{C}$  requires thorough sample preparation, including combustion or acid digestion, followed by radiochemical separation to remove interfering beta emitters.

New methods for in situ carbonaceous waste characterisation, although actual for the growing demand for decommissioning graphite reactors, have reached only TRL3 or TRL4. Until now in situ characterisation methods have been demonstrated in laboratory environments. There is a necessity to establish collaboration with the nuclear industry to adopt the developed measurement equipment for a user environment. The development of an integrated system of automatic sampling equipment and measurement equipment is also an actual task, which could enable the broader application of new equipment during decommissioning.

#### 8.3.2 $^{36}\text{Cl}$ main challenges

The methods based on LSC usually achieve good performance in terms of low MDA (10 mBq/g) and a sufficiently high chemical yield. Nevertheless, their complex and costly implementation poses a challenge to their widespread adoption in routine measurement activities. On the other hand, the methods relying on mass spectrometry still need further investigation to reach similar performance as LSC ones.

#### 8.3.3 $^{41}\text{Ca}$ main challenges

The determination of  $^{41}\text{Ca}$  is challenging due to very low activity concentration, extremely low energies of X-rays and Auger electrons in the range of LSC electronic noises (requiring a good separation from the matrix and the radionuclides emitting low energy electrons or X-rays), high stable Ca concentration interference in mass spectrometry and the lack of highly selective and simple separation procedures.

#### 8.3.4 $^{79}\text{Se}$ main challenges

For steel alloys, mass tailing from stable  $^{78}\text{Se}$  and  $^{80}\text{Se}$  are to be considered, and could pose a limit on achievable detection limits. Certified standard solutions of  $^{79}\text{Se}$  are not readily available, thus complicating a formal validation of radiometric measurement methods, for which assessment of detection efficiency must rely on radionuclides with similar energy.

#### 8.3.5 $^{93}\text{Zr}$ main challenges

The determination of  $^{93}\text{Zr}$  is challenging due to Zr chemistry (e.g. Zr tendency to hydrolyse), the lack of robust and standardised analytical procedures and of certified standard solutions [94]. To cope with the unavailability of certified  $^{93}\text{Zr}$ , the use of  $^{63}\text{Ni}$  could be more promising than  $^{95}\text{Zr}$  for the comparable energy range of the beta particles [92].

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#### 8.3.6 $^{93}\text{Mo}$ main challenges

Complex chemistry of molybdenum and lack of a certified standard solution of  $^{93}\text{Mo}$  are the main difficulties arising in its determination, thus hampering the formal validation of the laborious separation and measurement protocols.

#### 8.3.7 $^{99}\text{Tc}$ main challenges

The main challenges concerning the radiochemical separation of  $^{99}\text{Tc}$ , especially for the  $\beta$ -counting techniques, is their poor resolution. Considering mass spectrometric methods, such as ICP-MS, the challenges arise from interferences associated with matrix elements, which lead to isobaric or polyatomic interferences. Among these, the most significant ones derive from  $^{98}\text{Mo}$ - $^1\text{H}$  and  $^{99}\text{Ru}$  [123]. One suitable way to obtain Tc separated from Ru and Mo is through chromatography extraction using TEVA resin, but this method is much more expensive compared with anion exchange and solvent extraction [124]. It is therefore possible to observe that there are numerous techniques for separating and measuring  $^{99}\text{Tc}$ . However, these techniques are often expensive or time-consuming and should be simplified and validated for some specific waste matrices (e.g. spent ion exchange resins).

#### 8.3.8 $^{107}\text{Pd}$ main challenges

Main challenges for  $^{107}\text{Pd}$  determination are the complete removal of several interfering species and the lack of commercially available standard solution for method validation.

#### 8.3.9 $^{135}\text{Cs}$ main challenges

The need for high decontamination factors from interfering species and the lack of a certified standard solution of  $^{135}\text{Cs}$  are the main challenges for developing and validating robust separation and measurement protocols.

#### 8.3.10 $^{243/244}\text{Cm}$ main challenges

The complex determination of  $^{243}\text{Cm}$  and  $^{244}\text{Cm}$  activities is the main challenge for assessing and validating the SF of TRUs.

## 8.4 SF method limitations

The SF method has become a cornerstone for characterising DTM radionuclides in radioactive waste. However, implementation of this methodology faces several significant limitations. According to the IAEA publication on "Determination and Use of Scaling Factors for Waste Characterisation in Nuclear Power Plants," the SF method "relies on the establishment of a relationship between an easy-to-measure radionuclide (key nuclide) and DTM radionuclides," but this relationship often carries substantial uncertainty [17].

Kim et al. highlighted in their review of SF methodologies that current SF methods face challenges relating to statistical reliability, particularly when dealing with heterogeneous waste streams or when correlation data is limited [152]. The assumption of consistent ratios between key nuclides and DTM radionuclides breaks down in waste streams with varying operational histories, different contamination mechanisms, or heterogeneous compositions.

Research by Zaffora et al. demonstrated that conventional SF approaches can result in significant uncertainty in DTM activity estimations [19]. The authors proposed a Bayesian framework to update SF and reduce uncertainty, but noted that effective implementation requires substantial reference data that is often unavailable for many waste streams.

The ISO standardisation of the SF method (ISO 21238:2007) has improved methodological consistency but has not addressed fundamental limitations in applicability across diverse waste streams [13]. The implementation of standardised approaches still faces significant challenges when applied to heterogeneous waste packages or waste with complex contamination histories.

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The SF methodology should only be applied when a correlation between DTM and ETM has been established and proven to be representative for the waste stream. This requirement poses challenges in source sampling, radiochemical measurements, and data processing, which are not easy to implement when working with radioactive material, where sample collection and analysis are always subject to radiological protection constraints. For this reason, sampling optimisation processes, improvements in radiochemical techniques, and the combination of statistical analysis with theoretical isotope production models could enhance the reliability of the correlation model.

## 9. Summary & outlook

This report has provided a comprehensive state-of-the-art review of innovative characterisation techniques for large volumes of low and intermediate-level mixed radioactive waste generated during nuclear facility decommissioning. Through detailed analysis of NDT, DT, SF methodologies, and data management approaches, several key conclusions emerge.

The characterisation of radioactive waste represents a critical foundation for effective waste management throughout the entire lifecycle, from waste generation to final disposal. While significant advances have been made in both NDT and DT, important technical gaps remain that limit efficiency, accuracy, and cost-effectiveness of current methodologies.

Non-destructive characterisation techniques, including gamma spectrometry, neutron interrogation, and innovative imaging systems, have significantly progressed but face limitations in detection sensitivity for DTM radionuclides, processing speed, handling of non-standard geometries, and data analysis automation. The integration of artificial intelligence and machine learning approaches, while promising, remains largely untapped in operational settings.

DT provide essential complementary information, particularly for DTM radionuclides, but generate secondary waste and face challenges in sample representativeness, analysis time, and cost. The development of more efficient radiochemical separation methods and improved detection limits for key radionuclides remains a priority. Examples of limitations to be overcome are time-intensive sample preparation, complex matrix and interference removal, unsatisfactory detection limits, excessive analysis costs, absence of certified standard materials, lack of intercomparison exercises for method validation and performance assessment.

The SF methodology has emerged as a cornerstone approach for radioactive waste characterisation, bridging the gap between comprehensive radiochemical analysis and practical field implementation. The evolution of this methodology—from ISO's foundational approach to IAEA's expanded applications and PREDIS's collaborative innovation model—demonstrates a growing sophistication in statistical approaches, uncertainty quantification, and validation procedures. However, challenges remain in statistical reliability, particularly for heterogeneous waste streams or when correlation data is limited.

Digitalisation represents a transformative force in radioactive waste management, with digital twins, standardised data formats, and blockchain technology offering enhanced monitoring, predictive modelling, and decision-making capabilities. These technologies facilitate improved automation, uncertainty quantification, and regulatory compliance, though many remain at developmental stages rather than in routine operational use.

Looking forward, several key directions for future research and development emerge:

- **Enhanced Integration of NDT and DT Methods:** Developing systematic approaches to combine NDT and DT to optimise information value while optimising sampling requirements. This includes establishing more reliable correlations between field measurements and laboratory analysis to improve SF determination.
- **Advanced AI and Machine Learning Implementation:** Accelerating the adoption of AI and ML approaches for automated data interpretation, particularly for complex gamma spectra analysis, heterogeneity assessment, and predictive modelling of waste package behaviour.
- **Improved Field-Deployable Technologies:** Developing more sensitive, faster, and versatile field-deployable characterisation technologies that can handle diverse waste forms and container geometries, reducing the need for centralised characterisation facilities and supporting in-situ decision-making.

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- **Standardisation and Harmonisation:** Promoting international standardisation and validation of characterisation methodologies, data formats, and uncertainty quantification approaches to facilitate knowledge sharing across projects and national programs, particularly beneficial for countries with smaller nuclear programs.
- **Digital Transformation:** Advancing the implementation of digital twins, blockchain-based traceability, and integrated data management systems that connect characterisation data with waste management decisions throughout the entire waste lifecycle.
- **Cost-Efficiency Optimisation:** Developing graded approaches to characterisation that balance regulatory requirements with practical implementation constraints, ensuring that characterisation efforts are proportional to radiological risks and disposal route requirements. Developing simpler, quicker, and cheaper methods for the determination of DTM radionuclides
- **Knowledge Preservation:** Establishing robust systems for preserving characterisation data and methodological knowledge over the extended timeframes relevant to radioactive waste management, ensuring that future generations can interpret and utilise current characterisation information.

The effective characterisation of large volumes of radioactive waste remains a multidisciplinary challenge requiring continued innovation and collaboration between research institutions, regulatory bodies, and waste management organisations. WP5 ICARUS, by addressing the identified technical gaps and pursuing these future directions, can contribute to advancing toward more efficient, accurate, and cost-effective characterisation methodologies that support the safe and sustainable management of radioactive waste.

## Appendix A. Past RD&D projects on waste characterisation

- CHANCE project – Characterisation of conditioned radioactive waste, funding from Horizon 2020 Euratom Work Programme under grant agreement n° 755371, 2017–2021, <https://www.chance-h2020.eu/>.
- INSIDER project – Improved nuclear site characterisation for waste minimisation in decommissioning and dismantling operations under constrained environment, funding from the Euratom Research and Training Programme under grant agreement n° 755554, 2017-2021, <https://insiderh2020.eu/>.
- MICADO project – Measurement and instrumentation for cleaning and decommissioning operations, funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement n° 847641, 2019-2023, <https://www.micado-project.eu/>.
- PLEIADES – Platform based on Emerging and Interoperable Applications for enhanced Decommissioning processES, funding from Horizon 2020 Euratom Work Programme under grant agreement n° 899990, 2020-2023, <https://cordis.europa.eu/project/id/899990>.
- CLEANDEM project – Cyber physical equipment for unmanned nuclear decommissioning measurements, funding from Horizon 2020 Euratom Work Programme under grant agreement n° 945335, 2021-2024, <https://cordis.europa.eu/project/id/945335>.
- SHARE project – Creating a strategic plan for the research focused on enhancing safety, reducing environmental impact, and cutting costs in the decommissioning process, funding from European Union’s Horizon 2020 Research and Innovation Programme under grant agreement n° 847626, 2019-2022, <https://share-h2020.eu/>.
- PREDIS project – the development and implementation of activities for pre-disposal treatment of radioactive waste streams other than nuclear fuel and high-level radioactive waste, funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement No 945098 (2020-2024) <https://predis-h2020.eu/>



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**EURAD-2** Deliverable 5.1 – State-of-the-art on innovative non-destructive techniques, destructive techniques, scaling factors for use cases

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