D2: Guide on the use of mass spectrometry for low level radionuclide detection

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1 Summary

Mass spectrometry is increasingly being applied to the measurement of radioactivity in a range of areas including environmental monitoring, nuclear decommissioning and nuclear forensics. There are a range of mass spectrometric designs, which combined have published results for successful measurement of approximately 30 radionuclides. Given that the applications in this field are expected to increase further, it is timely to assess the major measurement considerations and potential future developments of this technique.

2 Scope

This document gives an overview of the role of mass spectrometry for radioactivity measurement and the major measurement considerations. The aim is for this guide to support the measurement of a range of sample matrices and radionuclides, for all applications including nuclear decommissioning, environmental monitoring, nuclear forensics and emergency response scenarios. The guide focuses on mass spectrometric techniques used in the MetroPOEM project for measurement of radioactive samples (ICP-MS and AMS). Other techniques (e.g. GDMS, SIMS, TIMS) are mentioned and there is the potential to extend this guide further. The relative advantages and limitations of mass spectrometric techniques are considered. There is no direct comparison between mass spectrometry and radiometric techniques.

3 Introduction

3.1 Importance of radionuclide measurement

Radionuclides are present in the environment as both naturally occurring (e.g. ^{40}K, ^{210}Pb, ^{226}Ra) and anthropogenic (e.g. transuranic Np, Pu, Am, Cm). In both cases, the concentration can vary significantly depending on the starting source and the environmental conditions. Regardless, accurate measurement is always of critical importance across several sectors.

Firstly, decommissioning of nuclear sites represents a significant global challenge, with high expense and the need to quantify various radionuclides in complex materials. Accurate measurement provides improved confidence that decommissioning wastes will be assigned to the appropriate waste category for subsequent storage or disposal. The incorrect classification of such wastes would have significant safety and economic implications.

Secondly, environmental monitoring of radioactivity contributes to the safety of the public and natural environment, such as measurement in air, drinking water and food.

Thirdly, identifying the source of a radioactive signal through isotopic ratio measurements is a valuable capability. This can be through identifying the source of nuclear material that has been illegally obtained, radiological dating, long-term monitoring of environmental patterns such as ocean currents, or as an indicator of a change in reactor operating conditions.

Nuclear medicine is a rapidly expanding area, where the production of the next generation of radiopharmaceuticals is offering more targeted and effective cancer treatment. To be effective, there must be confidence in the measurement of these radionuclides and removal of any impurities prior to patient administration.

In emergency response scenarios, there is a need to rapidly assess the radioactivity present and potential risks to the public and the environment.

Finally, all radionuclide measurements are underpinned by the understanding of the nuclear decay data and the half-life value, and there is on ongoing need to improve the accuracy of these for multiple radionuclides.

3.2   Role of mass spectrometry in radionuclide measurement

Mass spectrometry has long been established as a powerful analytical technique for measurement of most stable elements. Major application areas include quality control checks for sectors including pharmaceuticals; environmental measurement related to (for example) air and water quality; testing the purity of materials used in manufacturing such as semi-conductors.

Measurement of radionuclides by mass spectrometry represents a relatively small but growing area, with most measurements still carried out using decay counting techniques such as alpha spectrometry, liquid scintillation counting and gamma spectrometry. Mass spectrometry expands the number of radionuclides that can be measured compared to decay counting techniques alone. A key advantage is the rapid measurement of as low as several minutes per sample, with some setups able to process hundreds of samples per day and simultaneously measuring multiple radionuclides in the same run if needed.

Radionuclides are measurable by activity counting and mass spectrometric techniques, and it is possible to link the two methods (Lariviere, 2006). Radioactive decay occurs at a constant decay rate, 'A' as a function of the number of radioactive atoms (N) and the decay constant (λ) (Equation 1):

$$A = N\lambda \qquad \text{(Equation 1)}$$

If λ is known for a given radionuclide, the activity and mass can be linked based on N being proportional to the mass of the radionuclide (m) with respect to molar mass (M) and Avogadro's constant (N_A) (Equation 2):

$$N = \frac{N_A \times m}{MM} \qquad \text{(Equation 2)}$$

This can be rearranged for mass (m) by substituting Equation 1 into Equation 2 (Equation 3):

$$N = \frac{A \times MM}{N_A \times \lambda} \qquad \text{(Equation 3)}$$

Equation 3 can be rearranged for the half-life ($t_{1/2}$) given that $\lambda = \ln(2) / t_{1/2}$ (Equation 4):

$$m = \frac{A \times MM \times t_{1/2}}{ln2 \times t_{1/2}} \qquad \text{(Equation 4)}$$

If the half-life is expressed in seconds, activity in Becquerels (Bq) and molar mass in grams per mol (g/mol), the analyte mass will be in grams (g). Equation 4 shows that the ability of mass spectrometry to measure a radionuclide is primarily dependent on the half-life. The specific activity of a radionuclide (rate of radioactive decay for a given mass) is inversely proportional to the half-life. This means that, for longer-lived radionuclides, there are relatively more atoms to count for a given activity, favouring measurement by mass spectrometry. It is feasible to measure higher activities of shorter-lived radionuclides by mass spectrometry, however, this may necessitate modifying the instrument to handling higher activities (such as a glovebox), and decay counting techniques are considered better suited to such measurements. Examples of the impact of the half-life on mass and activity concentrations are shown in Table 1.

Based on the equations above, for a given radionuclide, the specific activity (SpA (Bq/g)) is calculated using equation 5:

$$SpA = \frac{ln2 \times N_A}{MM \times t_{1/2}}$$ (Equation 5)

The mass equivalent to 1 Bq (expressed in g) can be calculated using Equation 6:

$$\text{Mass equivalent to 1 Bq (g)} = \frac{1}{SpA}$$ (Equation 6)

Table 1. Example radionuclides showing the impact of half-life on mass/activity conversions

Radionuclide	Half-life / a	Specific Activity / (Bq/g)	Mass equivalent to 1 Bq / g
^{90}Sr	28.8 (7)	5.11×10^{12}	2.0×10^{-13}
^{63}Ni	98.7 (24)	2.13×10^{12}	4.7×10^{-13}
^{226}Ra	$1.600 (7) \times 10^3$	3.66×10^{10}	2.7×10^{-11}
^{239}Pu	$2.4100 (11) \times 10^4$	2.30×10^9	4.4×10^{-10}
^{99}Tc	$211.5 (11) \times 10^3$	6.33×10^8	1.6×10^{-9}
^{129}I	$16.1 (7) \times 10^6$	6.37×10^6	1.6×10^{-7}
^{238}U	$4.468 (5) \times 10^9$	1.24×10^4	8.0×10^{-5}

Early measurements of radionuclides by mass spectrometry focused on very long-lived radionuclides with limited interferences, such as ^{238}U (Halverson, 1987; Brown et al. 1988; Kubick et al. 1986). As the technique has evolved, the number of radionuclides and sensitivities achievable have improved, whilst maintaining the high sample throughput.

The ability of mass spectrometry to measure a radionuclide is determined by the half-life and the interferences that must be removed. For a given activity concentration, a radionuclide with a longer half-life will have more atoms to count and will be more sensitive than a shorter-lived radionuclide. As a result, mass spectrometry is today the preferred technique for multiple long-lived radionuclides, such as ^{Nat}U, ^{236}U, ^{237}Np and ^{239}Pu/^{240}Pu. In other cases, the number of applications using mass spectrometry compared to decay counting techniques is increasing, such as ^{90}Sr, ^{99}Tc and ^{129}I. In other cases, radionuclides that are very challenging to measure or not possible using decay counting techniques have been successfully detected by mass spectrometry, such as ^{59}Ni and ^{135}Cs. As the technique has advanced and the sensitivity and interference removal capabilities have improved, the half-life measurable even for low level applications has become shorter, with detection limits in the 10^{-18} g/g region achievable and radionuclides such as ^{210}Pb (22.23 (12) a) and ^{228}Ra (5.75 (4)) now measurable.

A list of radionuclides measurable by mass spectrometric techniques is shown in

Table 2. In some cases, detailed review papers have been published, which are referenced.

Table 2. Summary of radionuclides measured by mass spectrometry

Radionuclide	Half-life / a (DDEP where available)	Example of mass spectrometry technique used	References
^{14}C	5.70 (3) × 10^3	AMS	(Jiang et al. 2025; Linick et al. 1989; Skipperud and Oughton 2004)
^{32}Si	1.53 × 10^2	AMS	(Morgenstern et al. 2010; Schlomberg 2024)
^{36}Cl	302 (4) × 10^3	AMS, ICP-MS/MS	(Jiang et al. 2004; Llopart-Babot et al. 2023)
^{41}Ca	100.2 (17) × 10^3	AMS, RIMS, TIMS	(Xia et al. 2023; Hampe et al. 2013; Jorg et al. 2012)
^{59}Ni	76 (5) × 10^3	AMS	(Grestis and Tolgyesi 2004)
^{63}Ni	98.7 (24)	ICP-MS/MS	(Russell et al. 2024)
^{79}Se	356 (40) × 10^3	AMS, MC-ICP-MS	(Wang et al. 2010; Comte et al. 2003)
^{90}Sr	28.8 (7)	ICP-MS/MS, ICP-SFMS, AMS, MC-ICP-MS	(Martschini et al. 2022; Zhou et al. 2023; Ohno et al. 2018; Isnard et al. 2006)
^{93}Zr	1.61 (6) × 10^6	ICP-MS/MS, MC-ICP-MS	(Petrov et al. 2017; Asai et al. 2018)
^{93}Mo	4.0 (8) × 10^3	ICP-MS/MS	(Tanaka et al. 2024)
^{99}Tc	211.5 (11) × 10^3	ICP-MS/MS, ICP-QMS	(Shi et al. 2012)
^{107}Pd	6.50 × 10^6	ICP-MS	(Faure et al. 2024)
^{126}Sn	1.00 × 10^5	ICP-QMS	(Asai et al. 2013)

^{129}I	16.1 (7) × 10^6	ICP-MS/MS, AMS	(Matsueda et al. 2022; Zhang et al. 2021)
^{135}Cs	2.3(3) × 10^6	ICP-MS/MS, AMS	(Russell et al. 2015; Cao et al. 2016)
^{151}Sm	94.7 (6)	ICP-MS/MS, MC-ICP-MS	(Garcia Miranda et al. 2018; Be et al. 2015)
^{210}Pb	22.23 (12)	MC-ICP-MS, AMS	(Kristensen and Hou, 2025; Bashkaran et al. 2018; Vivo-Vilches et al. 2021)
^{226}Ra ^{228}Ra	1.600 (7) × 10^3 5.75 (4)	ICP-SFMS, MC-ICP-MS, ICP-MS/MS	(Boudias et al. 2024; Dalencourt et al. 2018; Hsieh and Henderson 2011)
^{229}Th ^{230}Th ^{232}Th	7.88(12) × 10^3 75.38 (30) × 10^3 14.02 (6) × 10^9	ICP-MS, ICP-MS/MS, MC-ICP-MS, ICP-SFMS, TIMS	(Aggarwal, 2016; Varga et al. 2025)
^{237}Np	2.144 (7) × 10^6	ICP-MS, ICP-MS/MS, MC-ICP-MS, ICP-SFMS	(Thakur, 2012)
^{232}U ^{233}U ^{234}U ^{235}U ^{236}U ^{238}U	70.6 (11) 159.1 (2) × 10^3 245.5 (6) × 10^3 704 (1) × 10^6 23.43 (6) × 10^6 4.468 (5) × 10^9	ICP-MS, ICP-MS/MS, MC-ICP-MS, ICP-SFMS, ICP-TOF-MS, TIMS	(Steier et al. 2008; Santos et al. 2010; Scott et al. 2024)
^{239}Pu ^{240}Pu ^{241}Pu ^{242}Pu ^{244}Pu	24.110 (11) × 10^3 6.561 (7) × 10^3 14.33 (4) 373 (3) × 10^3 81.1 (6) × 10^6	ICP-MS, ICP-MS/MS, MC-ICP-MS, ICP-SFMS, ICP-TOF-MS, TIMS	(Steier et al. 2013; Hou et al. 2019; Hotchkis et al. 2019; Metzger et al. 2019)
^{241}Am ^{243}Am	432.6 (6) 7.67 (23) × 10^3	ICP-SFMS, MC-ICP-MS	(Aggarwal, 2016; Mathew et al. 2020)

As well as single radionuclide measurement, isotopic ratio measurements are a powerful tool for determining the source of radioactivity e.g. weapons fallout, discharges from nuclear fuel reprocessing facilities, or nuclear accidents such as Chernobyl or Fukushima. In some cases, these isotopic ratios can only be measured by mass spectrometry and not by decay counting techniques.

For example, $^{135}Cs/^{137}Cs$ is increasingly being demonstrated as a powerful forensic tool, with long-lived ^{135}Cs offering a good alternative to short-lived ^{134}Cs that has been used as a forensic tool through $^{134}Cs/^{137}Cs$ measurement. The atomic ratio of $^{135}Cs/^{137}Cs$ is close to 1 in fresh nuclear fission waste, but the difference in half-life makes the activity ratio closer to 1 : 80,000, making determination of ^{135}Cs by decay counting techniques challenging. Comparatively, mass spectrometry can simultaneously measure both ^{135}Cs and ^{137}Cs. Examples of isotopic ratios measurable by mass spectrometry are shown in Table *3*. In some cases, the ratios can be two radioactive isotopes, in others (such as $^{41}Ca/^{40}Ca$ and $^{129}I/^{127}I$) it is a combination of stable and radioactive isotopes. The range of isotopic ratios measurable depends on the application area as well as the dynamic range and abundance sensitivity of the detector. In some cases (e.g. $^{41}Ca/^{40}Ca$, measurement of ratios $<10^{-12}$ may be required, which is only achievable by highly sensitive techniques such as AMS or RIMS.

Table 3. Examples of isotopic ratios measurable by mass spectrometry

Isotopic ratio	References
$^{41}Ca/^{40}Ca$	(Xia et al. 2023; Hampe et al. 2013; Jorg et al. 2012)
$^{129}I/^{127}I$	(Matsueda et al. 2022; Zhang et al. 2021)
$^{135}Cs/^{137}Cs$	(Russell et al. 2015; Cao et al. 2016)
$^{226}Ra/^{228}Ra$	(Boudias et al. 2024; Dalencourt et al. 2018; Hsieh and Henderson 2011)
$^{234/235/236/238}U$	(Santos et al. 2010; Scott et al. 2024)
$^{239}Pu/^{240}Pu$	(Steier et al. 2013; Hou et al. 2019; Metzger et al. 2019)

3.3   **Overview of mass spectrometry**

3.3.1   Major components

There are three major components to a mass spectrometer, with variations in design amongst all of them.

1. The ion source converts the sample into ions, which are extracted and passed through a mass analyser.
2. The mass analyser separates ions by their mass-to-charge ratio before they reach the detector.
3. The detector provides an output that can be used to calculate the abundance of the ions present.

There are a range of mass spectrometer designs, summarised in **Fehler! Verweisquelle konnte nicht gefunden werden.**. Inductively coupled plasma mass spectrometry (ICP-MS) is perhaps the most common, with a range of configurations. This review does not describe the instrument layout in detail or include every radionuclide measurable by each technique, but, where available, review articles on the use of specific techniques for radionuclide analysis have been included in **Fehler! Ungültiger Eigenverweis auf Textmarke.**.

Table 4. Summary of mass spectrometry designs

Acronym	Full name	References
ICP-QMS	Inductively coupled plasma quadrupole mass spectrometry	(Hou and Per Roos, 2008; Croudace et al. 2017; Kutschera et al. 2023; Quemet et al. 2024)
ICP-MS/MS	Tandem inductively coupled plasma quadrupole mass spectrometry	
ICP-SFMS	Sector field inductively coupled plasma quadrupole mass spectrometry	
MC-ICP-MS	Multi-collector inductively coupled plasma quadrupole mass spectrometry	
ICP-TOF-MS	Inductively coupled plasma time of flight mass spectrometry	
AMS	Accelerator mass spectrometry	
TIMS	Thermal ionisation mass spectrometry	
RIMS	Resonance ionisation mass spectrometry	
SIMS	Secondary ionisation mass spectrometry	
SNMS	Sputtered neutral mass spectrometry	

Prior to ionisation, the way in which the sample is introduced varies significantly with instrument design and can impact the sensitivity and extent of interference removal.

For some mass spectrometric designs such as ICP-MS, there is flexibility in the sample introduction prior to the ion source depending on the measurement requirements. The reasons for this flexibility could be:

- Sample type – such as laser ablation for direct measurement of solid samples, gas chromatography for gaseous samples, or various solution-base systems for aqueous samples.

- Sensitivity- some setups offer enhanced sensitivity for very low-level measurement. These may be more sensitive to matrix-effects and therefore only suitable for low-level samples following extensive sample preparation.

- Amount of sample- when the amount of sample available is low, there are sample introduction systems that have very low uptake rates on the order of µL/minute, or nebulisers that use all of the sample and do not lose any to waste.

- Interference removal- this can include desolvating sample introduction that reduces the water loading into the instrument, resulting in lower hydride and oxide polyatomic interference formation. Alternatively, chemical separation instruments can be coupled directly to the sample introduction to automate separation and measurement.

- Sample matrix- specifically for aqueous samples, certain samples matrices may need specific sample introduction systems, such as the addition of an Argon/Oxygen mix for organic samples, or a Perfluoro alkoxy alkane (PFA) system for samples containing hydrofluoric acid.

- Volatility- volatile radionuclides such as ^{129}I may be limited to certain sample introduction systems. For example, the desolvating sample introduction system mentioned previously heats the sample, which would result in iodine losses.

Some examples of sample introduction systems for ICP-MS are given in Table *5*.

Table 5. Examples of sample introduction system for ICP-MS

Sample introduction	Comments	References
Solution	Range of designs and matrix tolerances; relatively high solvent loading	Boulyga and Becker, 2002; Becker and Dietze, 1999)
Desolvating	Reduced solvent loading leading to reduced oxide and hydride formation in the plasma; improved sensitivity	(Minnich and Houk, 1998; Zheng, 2015)
Sequential injection	Automated analysis through online chemical separation directly injected into the ICP	(Qiao et al. 2010)
Laser ablation	Direct measurement of solid samples as single particles, surface or depth profiling	(Chen et al. 2014; Becker, 2005)
Electrothermal vaporisation	Sample introduced into a heated tube or filament, sample material vaporised into a stream of carrier gas over the furnace before recondensing; can handle complex matrices, potentially higher detection limits	(Truscott et al. 1999)
Glow discharge	Voltage applied across two electrodes separated by low pressure gas; gas ionised by electric field	(De las Heras et al. 2022)
High Performance Liquid Chromatography (HPLC)	Automated analysis- separated sample directly introduced into the ICP	Gunther-Leopold et al. 2005)

The ion source, and therefore the way the sample is prepared for measurement, varies with instrument design.
This is summarised in

Table *6*.

Table 6. Examples of ionisation sources in different mass spectrometers

Mass spectrometer	Ionisation source
ICP-MS (all)	High temperature Argon plasma to generate positive ion beam
AMS	Solid target injected by ion sputtering to generate negative ion beam
TIMS	Aqueous volume deposited and dried on a filament (mostly high purity Re) which is evaporated and ionised
RIMS	Solid or liquid sources vaporised and atomised on atomic beam source e.g. evaporation of sample using electron beam
SIMS	Solid samples sputtered by bombardment with focused primary ion beam and sputtered secondary ions measured
SNMS	Separation of ionisation from sputtering; electron impact ionises sputtered neutral material and secondary ions deflected from analyser

Some designs (such as LA-ICP-MS and secondary ionisation mass spectrometry (SIMS)) allow for direct measurement of solid samples without prior sample preparation. Other techniques that require sample preparation prior to measurement could result in a longer analytical procedure, but very low detection limits can be achieved.

For ICP-MS, despite the range of sample introduction options, the ion source is consistently a high temperature (approximately 7000 K) argon plasma to decompose samples and produce a beam of positively charged ions.

By comparison, samples for AMS are prepared as a solid target and injected by ion sputtering (commonly using a Cs^+ ion source) to generate a negative ion beam. Samples are mounted onto a wheel or other movable device to be inserted into the ion source sample changing mechanism. There are several different designs. An important factor is the centring of the mechanism, to guarantee best impact of the Cs^+ sputter beam in creating negative ions for extraction. An off-centre beam negatively impacts overall sample usage (and thus sensitivity!), and further both long and short-term output stability.

Depending on the element and the type of ion source there is a risk of crosstalk between samples in the ion source. The risk is highest for volatile elements such as iodine. Expected differences in the concentration of samples to be sputtered in the same measurement therefore must be considered when preparing the sample magazine/wheel.

3.4 Sample preparation

3.4.1 Role of sample preparation

The role of sample preparation is to prepare the radionuclide(s) of interest for measurement. The extent and nature of sample preparation varies depending on several factors, including:

- Radionuclide(s) of interest – for example, volatile radionuclides such as ^{99}Tc or ^{129}I must be handled differently to non-volatile radionuclides to prevent significant loss during preparation.

- Starting activity concentration- if the starting activity concentration is expected to be above the mass spectrometers limit of detection for the radionuclide(s) of interest, it may be possible to perform the measurement with little or no sample preparation.

- Sample matrix- more complex sample matrices such as concrete or soil may require multiple sample preparation stages to remove interferences and/or get the material into a form where measurement is possible. In other cases, dilution to reduce the impact of the sample matrix may be sufficient.

- Interferences- different radionuclides and sample matrices will contain varying levels of interferences, which impacts the extent of sample preparation, specifically offline and/or online chemical separation depending on the mass spectrometer being used.

- Mass spectrometric technique- depending on the sample introduction method and the online interference removal capabilities, any sample preparation must reach an end point where the sample is suitable for introduction to the mass spectrometer being used.

The aim is to prepare a sample such that sufficient recovery and purity of the radionuclide(s) is achieved for mass spectrometric measurement.

3.4.2 Reference standards, materials and tracers

3.4.2.1 Overview and production challenges

The increasing application of mass spectrometry for measuring a growing number of radionuclides must be supported by the availability of calibration standards, tracers and reference materials to support measurements.

Reference materials are valuable in validating the performance of a method for radionuclide measurement. Such materials can be spiked, synthetic or 'real' materials from a selected environment.

A tracer is an isotope of the radionuclide of interest that is added to the sample at the start of the procedure to assess the chemical yield during sample preparation, such that any losses can be corrected for.

Calibration standards are used to assess the instrument sensitivity using traceable standards of the radionuclide(s) of interest.

There is limited availability of reference materials, tracers and calibration standards for multiple radionuclides that are needed to support mass spectrometry measurement. Amongst the aims of the MetroPOEM project were to develop such standards and reference materials to help address this need.

In all cases, it is a time-consuming and analytically challenging process to produce and characterise standards, tracers and reference materials. Amongst the challenges are preparing standards and reference materials containing the appropriate radionuclides for end users at appropriate activity levels. In the case of highly sensitive techniques such as AMS, very low-level standards are needed that are challenging to prepare. There is unlikely to be a consensus on a single material or set of standards that are required given the range of applications between laboratories. The development of any standards and materials is generally of high value to the community. To overcome the shortage, some laboratories use internally available materials that are less available or too valuable for routine measurements.

In the case of tracers, the development of mass-spectrometric radionuclide tracers has perhaps not kept up with the number of radionuclides that can be measured. Ideally, a tracer would not be present in the sample and have a suitably long half-life that could also be measured by mass spectrometric techniques, such as ^{233}U for natural U isotopes or ^{242}Pu for ^{239}Pu and ^{240}Pu. In some cases, radionuclides do not have a long-lived tracer available or use a shorter-lived tracer that must be measured using a decay counting technique, such as ^{239}Np

for [237]Np or [223]Ra for [226]Ra. The use of a stable tracer of the same element as the radionuclide may be possible, however, the concentration of this stable element in the sample must be known such that a suitable level of tracer can be added, which in turn must not interfere with the measurement.

Table *8* summarises the radioactive tracers available and the measurement techniques available to assess them.

Table 7. Examples of radioactive tracers available for radionuclides measurable by mass spectrometry, as well as potential candidate tracers that would be suitable for mass spectrometry that are not currently available. Stable isotope tracers have not been included.

Radionuclide	Current radioactive tracer	Tracer half-life	Measurement technique	Candidate radioactive tracer for mass spectrometry (if relevant)	Candidate tracer half-life
[14]C	-	-	-	-	-
[32]Si	-	-	-	-	-
[36]Cl	-	-	-	-	-
[41]Ca	[45]Ca	162.64 (11) d	Gamma spectrometry	-	-
[59]Ni, [63]Ni				-	-
[90]Sr	[85]Sr	64.850 (7) d	Gamma spectrometry		

^{99}Tc	^{95}Tc		Gamma spectrometry	^{97}Tc ^{98}Tc	
^{129}I	^{125}I	59.388 (28) d	Gamma spectrometry	-	-
^{135}Cs/^{137}Cs	^{134}Cs	2.0644 (14) a	Gamma spectrometry	-	-
^{151}Sm	^{147}Sm	107.9 (12) × 10^9 a	Mass spectrometry	-	-
^{210}Pb	^{205}Pb	17.3 (7) × 10^6 a	Mass spectrometry		
^{226,228}Ra	^{223}Ra	11.43 (3) d	Gamma spectrometry	-	-
^{230,232}Th	^{229}Th	7.88 (12) × 10^3 a	Mass spectrometry Alpha spectrometry	-	-
^{237}Np	^{239}Np	2.356 (3) d	Gamma spectrometry	^{236}Np	155 (8) × 10^3
^{234,235,236,238}U	^{232}U ^{233}U	70.6 (11) a 159.1 (2) × 10^3 a	Mass spectrometry Alpha spectrometry	-	-
^{239,240,241}Pu	^{242}Pu	373 (3) × 10^3 a	Mass spectrometry Alpha spectrometry	-	-
^{241}Am	^{243}Am	7.367 (23) × 10^3 a	Mass spectrometry Gamma spectrometry	-	-

3.4.2.2 Measurement considerations

Isotope reference materials or in house secondary standards must be traceable to the SI; overviews of existing materials are published elsewhere (Brand et al., 1979).

For an AMS system it is not possible to infer mass bias effect completely from first principles to the degree needed in high precision measurements. Consequently, calibration standards or internal reference materials are measured in regular intervals to allow for normalisation of the raw ratios. To track machine stability (stability of the mass bias correction) a set of samples (e.g. 5-8 samples + 1 processing blank) is usually bracketed between measurement of standards. Usually too long measurement time spend on an individual run, instead the sequence is repeated with shorter runs. As negative ion formation and beam extraction strongly depends on impurities still present in the AMS sputter target, it is important to verify the overall analytical procedure starting from the sample preparation with representative reference materials (same material, similar concentration and mass). It is recommendable to include a corresponding reference material in each measurement for quality control.

3.4.3 Interference removal

As with radiometric techniques, accurate measurement of radionuclides by mass spectrometry is dependent on effective removal of interferences. The main interferences to consider are:

- Isobaric - a single isotope with a similar mass to the radionuclide of interest. This can be a stable isotope e.g. ^{90}Zr and ^{90}Sr, or radioactive e.g. ^{241}Am and ^{241}Pu.

- Polyatomic - two or more isotopes with a combined mass similar to the radionuclide of interest. For example, splitting of water in the high temperature plasma in ICP-MS can lead to hydride (H) and oxide (O) interferences, leading to interferences such as $^{74}Ge^{16}O$ and $^{89}Y^{1}H$ on ^{90}Sr. Other elements than can form polyatomic interferences include Argon (used as a plasma gas), carbon and nitrogen.

- Tailing - an isotope one or two mass units either side of the radionuclide of interest with a significantly higher concentration. This can be from an isotope of a different element to the radionuclide, such as ^{238}U that affects both ^{237}Np and ^{239}Pu, or from an isotope of the same element, such as ^{88}Sr on ^{90}Sr.

A summary of interferences that impact mass spectrometric measurement of radionuclides is shown in

Table *8*. The removal of isobaric interferences can be achieved by offline chemical separation prior to measurement and/or online separation such as collision/reaction cells. For tailing, if the interference is from an isotope of the same element, this cannot be removed by chemical separation and is dependent on the resolving power of the mass spectrometer used, which varies depending on the instrument design.

The impact of the sample matrix on measurement must also be considered. For example, seawater contains a high amount of total dissolved solids that can impact ion transmission and the signal. As well as effective sample preparation, the availability of suitable matrix-matched reference materials is important in quantifying and potentially correcting for any matrix effects.

Mass spectrometry can potentially be used to assess the initial interference level in the starting sample, which can be used to inform the extent of sample preparation required.

Table 8. Examples of interferences that affect radionuclide measurement by mass spectrometry.

*Shorter-lived radionuclides (half-life <1 a) have not been considered as isobars.

** There are an extensive range of polyatomic interferences, a couple of examples are given in each case.

***Isotopes identified in the literature as significant tailing interferences have been included.

Radionuclide	Isobar*	Polyatomic examples**	Tailing***
^3H	-	-	-
^{14}C	^{14}N	$^{13}C^1H$	^{13}C, ^{14}N
^{32}Si	^{32}S	$^{16}O_2$	^{31}P
^{36}Cl	^{36}Ar, ^{36}S	$^{35}Cl^1H$, $^{18}O_2$	^{35}Cl, ^{37}Cl
^{41}Ca	^{41}K	$^{40}Ca^1H$, $^{40}K^1H$	^{40}Ca, ^{40}K
^{59}Ni	^{59}Co	$^{58}Ni^1H$, $^{27}Al^{16}O_2$, $^{43}Ca^{16}O$	^{58}Ni
^{63}Ni	^{63}Cu	$^{62}Ni^1H$, $^{47}Ti^{16}O$	^{62}Ni, ^{64}Ni
^{90}Sr	^{90}Zr	$^{88}Sr^1H_2$, $^{89}Y^1H$, $^{74}Ge^{16}O$, $^{58}Ni^{16}O_2$	^{88}Sr
^{99}Tc	^{99}Ru	$^{98}Mo^1H$, $^{67}Zn^{16}O_2$	^{98}Mo
^{129}I	^{129}Xe	$^{97}Mo^{16}O_2$, $^{127}I^1H_2$	^{127}I
^{135}Cs	^{135}Ba	$^{133}Cs^1H_2$, $^{119}Sn^{16}O$	^{133}Cs
^{137}Cs	^{137}Ba	$^{121}Sn^{16}O$, $^{136}Xe^1H$	^{138}Ba
^{151}Sm	^{151}Eu	$^{150}Sm^1H$, $^{135}Ba^{16}O$	^{150}Sm
^{210}Pb	-	$^{208}Pb^1H_2$	^{208}Pb
^{226}Ra ^{228}Ra	- -	$^{88}Sr^{138}Ba$, $^{208}Pb^{18}O$ $^{196}Pt^{16}O_2$	- -
^{229}Th ^{230}Th ^{232}Th	^{232}U - -	- - -	- ^{232}Th -
^{237}Np	-	-	^{238}U
^{232}U	^{232}Th	-	-

^{233}U	-	$^{232}Th^{1}H$	^{232}Th
^{234}U	-	-	^{235}U
^{235}U	-	-	-
^{236}U	-	$^{235}U^{1}H$	^{236}U
^{238}U	-	$^{236}U^{1}H_{2}$	-
^{239}Pu	-	$^{238}U^{1}H$	^{238}U
^{240}Pu	-	$^{239}Pu^{1}H$	^{238}U
^{241}Pu	^{241}Am	$^{240}Pu^{1}H$	-
^{242}Pu	-	-	-
^{244}Pu	^{244}Cm	-	-
^{241}Am	^{241}Pu	$^{240}Pu^{1}H$	-
^{243}Am	-	-	-

3.4.4 Sample digestion

Mass spectrometry has effectively measured a range of radionuclides in various natural and anthropogenic solid matrices including seaweed, soil, steel, sediment, graphite and concrete. Direct measurement of solid samples is possible, for example through laser ablation-based sample introduction. Dissolution of the solid material is commonly performed prior to chemical separation of the radionuclide and/or measurement.

There are several major sample dissolution techniques available. The approach used depends on factors including the sample matrix, starting sample mass, radionuclides present and their interaction with the solid material e.g. sorbed to the surface of the material or more strongly held in the sample matrix, and the post-dissolution treatment e.g. direct measurement by mass spectrometry or radiochemical separation. Some options for sample dissolution are summarised in Table *9*.

Table 9. Examples of solid sample dissolution techniques.

Digestion technique	Comments	References
Acid digestion – HCl and HNO_3	High sample masses can potentially be processed; analyte recovery may be limited	(Bock, 1979; Russell et al. 2015)
Acid digestion – HF and $HClO_4$	More hazardous material; high analyte recovery achievable; fluorides must be removed by subsequent chemical separation	(Parsa, 1992)
Alkali fusion	Effective dissolution of complex matrices; potentially long treatment times; complex dissolved matrix	(Galindo et al, 2007)
Borate fusion	Effective dissolution of complex matrices; complex dissolved matrix	(Croudace et al. 1998)

For open vessel acid digestion, high starting masses can be handled, and depending on the acid used, complete dissolution can be achieved e.g. using hydrofluoric acid, although the use of these more hazardous acids must be considered. Microwave-assisted digestion offers close control of the temperature and pressure conditions to aid dissolution and improve reproducibility but can only handle a limited amount of sample in each microwave vessel (up to approximately one gram).

Fusion using various flux mixtures such as lithium borate or alkali fusion has been proven to be effective for complete dissolution of challenging matrices and situations where radionuclides are strongly held in the sample. The dissolved sample and flux mixture is relatively matrix-heavy compared to other techniques and will need to be considered for post-dissolution treatment.

3.4.5 Chemical separation

Chemical separation includes pre-concentration and purification to concentrate the target radionuclides and remove as much as possible interfering isotopes. The extent of the chemical separation procedure applied depends on the sample matrix, target radionuclide and extent of interferences.

Separation options include 'bulk' procedures such as precipitations, which are not radionuclide specific but can cope with complex sample matrices such as solid digests. Ion exchange chromatography is charge-specific and has a relatively high matrix tolerance. Extraction chromatography using resins from Triskem International are commonly used for radionuclide-specific separations (Triskem International, 2025). The ability of these resins to separate radionuclides from mass spectrometric interferences is increasingly being considered, whereas historically information was only provided for radiometric interferences. The matrix tolerance of extraction chromatography resins is relatively low compared to ion exchange or bulk separation techniques, which can be overcome by increasing the amount of resin used and/or preceding this with other chemical separation stages.

As chemical separation is accompanied by loss of the radioanalyte, this needs to be monitored and corrected for. This can be achieved using:

- A suitable stable reference isotope of the same element already present in the original sample matrix (e.g. ^{88}Sr for measurement of ^{90}Sr).

- Addition of a carrier (e.g. ^{9}Be for measurement of ^{10}Be).

- Addition of another isotope spike (e.g. ^{242}Pu for other Pu isotopes).

Depending on the carrier or isotopic spike available, it should be noted that a different method may be required to determine the reference isotope content in the matrix. This was discussed in the 'Reference Materials and Tracers' section.

3.4.6 Preparation of samples for measurement

3.4.6.1 ICP-MS

For ICP-MS, most applications are based on aqueous sample introduction, with samples typically prepared in dilute nitric acid (approximately 0.1-0.5 M HNO$_3$). Depending on the instrument sensitivity, radionuclide being measured and sensitivity needed, higher purity sub-boiled HNO$_3$ is recommended. In the case of volatile radionuclides such as ^{129}I, samples should be prepared in dilute alkali solutions such as sodium hydroxide.

3.4.6.2 AMS

For AMS, preparation is necessary to chemically prepare the sample as a sputter sample that is suited to put out negative ions in a Caesium sputter ion source. Typically for metals molecular compounds oxides, hydrides, and fluorides yield the best results, for some elements the atom forms negative ions readily (e.g. Carbon, Silicon, Chlorine, Iodine). In most cases, the radionuclide of interest has to be embedded in few mg of solid material. This can be the stable element extracted from the sample itself, e.g. graphite for ^{14}C measurements or a carrier of the same element added to the sample at the beginning of sample preparation, e.g. stable

Chlorine for ^{36}Cl measurements. If there are no stable isotopes, the radionuclide is embedded in a suitable matrix added after the chemical purification.

It is essential to ensure that no macroscopic quantities of other elements either from the initial sample or salts formed during sample preparation (e.g. oxalates, ammonium compounds) are present in the final AMS sample, also because of the limited volume of the AMS sample holder. Sample preparation in AMS can also help to reduce stable isobar interferences.

The matrix of the sample is usually mixed with an electrically conduction binder to avoid the impact of an electrically charging sample in the ion source. For actinide measurements the current standard sample preparation is to prepare the sample as oxide by co-precipitation in iron oxide (typically 2 mg). This is then mixed with Niobium as binder which has proven best for ion output. To avoid any additional impurities, the binder can be left out and the iron oxide pressed directly into the sample holder. This should be especially considered for samples with low ^{238}U content if U isotopes are to be measured relative to ^{238}U (e.g. peat or ice core samples). For Sr the sample is prepared as fluoride and then mixed with Pb fluoride. Copper is not to be used for sample holders and/or sample wheel for actinide oxide measurements as Cu forms clusters with similar mass. Related to this, Al sample holders and/or wheel are not to be used as fluoride compounds reacts with Al and migrate into the material.

3.5 Measurement procedure

3.5.1 Background

Blanks are required to track contamination in sample preparation, cross-contamination and ambient contamination in machine operation, possible base-level of a carrier material, and generic machine background events independent of contamination and samples. The background from the blanks needs to be factored into the measurement result, however, the treatment may differ due to the origin and isotopic composition of the background.

3.5.1.1 ICP-MS

Instrument blanks and procedure blanks are typically measured and subtracted from the real sample measurement. The purity of the reagent used for preparing the background depends on the sensitivity of the instrument, the radionuclide(s) being measured and their interferences, and the detection limits required.

For single radionuclide measurement, a blank is often measured after every sample. Instruments with autosamplers commonly have a recirculating blank reservoir that cleans the autosampler probe between each run. This reduces the risk of contamination compared to a 'static' blank solution, and in some cases the length of this cleaning cycle can be set to run until a threshold background count rate is achieved. Depending on the radionuclide and the activity level, several cleaning stages using different reagents may be required.

When performing isotope ratio measurements, in case of isotope dilution mass spectrometry (IDMS), when correctly performed, and when the procedure blank, the sample and the reference have a similar isotopic composition, blank correction is not necessary and if applied may lead to a bias or even wrong result. Usually monitoring the blank at the beginning and at the end of an IDMS experiment is sufficient.

3.5.1.2 AMS

If the source of contamination has a much larger isotopic ratio than the typical samples, the isotope ratio measured on the blanks can be simply subtracted from the sample isotope ratios if the reference isotope is the same within uncertainties between the samples and the blanks. Failing that, a per sample rare isotope absolute contamination amount must be determined from the blank samples, which is then subtracted from the uncorrected absolute rare isotope content inferred for the samples from the measured isotopic ratio.

If the source of blank contamination is similar to sample isotopic ratios, correction is more involved as the correction depends on both the sample and the blank ratio. For unknown ambient contamination processing of both blank and standard samples is required. This approach is followed in radiocarbon AMS.

The machine background is usually a constant per measurement time background and may be controlled by a special machine background sample free of the isotopes of interest, but similar in the remaining composition

(e.g. iron + niobium for actinide isotopes). This background is rarely relevant but should be monitored for samples with low ^{238}U content if $^{236}U/^{238}U$ is to be analysed. Cross-contamination in the ion source necessitates monitoring blank samples throughout the measurement period, whilst the base rare isotope level of a carrier material is usually small.

Blank levels considered for correction can either be global or local. Global blank levels are determined over time in the measurement of many blank samples and barring any unusual outliers, no consideration is given to individual batches of samples processed. This the practice in radiocarbon AMS. Local blanks refer to blank samples being processed together with sample batches and used only for those individually. This is commonly used for most other isotopes. Standards or internal reference materials are measured and their raw ratios versus their nominal ratios are used to determine the normalization ("correction factor", "mass bias correction") to convert measured ratios into "true ratios" of the sample or blank, correspondingly.

3.5.2   Internal standard

During measurement, there is the potential for the instrument performance to change due to variations in operating conditions e.g. argon gas supply to the plasma in ICP-MS, or through matrix effects from the samples being run. Mass spectrometry can run a high number of samples and some variation in performance during the run is to be expected.

An internal standard monitors for changes in instrument performance during a run. A weighed amount of internal standards can be added to every sample prior to measurement, with any difference in mass between samples accounted for. The first sample represents the 'time zero' measurement against which the other samples are assessed.

There are some instruments that offer online measurement of an internal standard, where a solution is constantly introduced to the instrument along with the sample. This avoids the need to manually add an internal standard to every sample.

There are several criteria for the element used as an internal standard. Firstly, it should be an element that has a similar ionisation efficiency to the radionuclide being measured. Some elements (such as Iodine) have a high first ionisation energy (10.45 eV) that impacts the sensitivity of radionuclides such as ^{129}I. An internal standard with a low first ionisation energy will therefore not be representative of ^{129}I when assessing changes in instrument conditions. The internal standard should also not be present in the samples being measured and not interfere with measurement of the radionuclide such as through polyatomic or tailing interferences. Related to this, the concentration used should be suitable for the instrument used to achieve good sensitivity.

Depending on the number, mass range and ionisation energies of the radionuclides being measured, more than one element can be used as an internal standard. Some instruments have software capabilities that can assign specific elements as internal standards and apply a correction.

Internal standards can be supported through the measurement of reference standards and quality control samples during a run.

3.5.3   Instrument tuning

Tuning of the instrument is of critical importance for optimising analyte sensitivity, minimising background and, depending on the instrument design, minimising interferences.

3.5.3.1   ICP-MS

Some modern instruments can automatically tune the instrument using criteria set by the manufacturer or the user. If the instrument is used for a range of applications, having a general tuning procedure is valuable to show that the baseline performance is acceptable.

Customised tuning of the instrument can significantly improve performance by focusing on the radionuclide(s) of interest. A suitable calibration standard is valuable, either of the radioactive element being measured or a stable analogue. Tuning of every component of the instrument is possible. This includes the sample introduction system, such as the physical positioning of the torch and the argon gas flow rates. The voltages of the various lenses that focus the ion beam to the mass analyser can be optimised.

The detector can also be tuned, which is particularly important for isotope ratio measurements and benefits from an isotopic ratio standard being available.

Typical parameters assessed when tuning ICP-MS include the sensitivity, background, oxide formation rate, doubly charged ion formation and peak axis settings.

For instruments with a collision-reaction cell, the tuning can include the cell gas flow rate and lens voltages to optimise the analyte signal and minimise interference contribution. This can be achieved through introducing a mixed standard containing the radionuclide and interferences.

3.5.3.2 AMS

Tuning for AMS involves more beam guiding elements than is typical for other mass spectrometers. As a result, considerable effort needs to be put into tuning to achieve optimum transmission across the masses of interest, thus minimising mass bias and spectrometer induced scatter of isotope ratio results. Typically, pilot beams of a reasonably abundant isotope measurable in Faraday cups (e.g. ^{238}U for most actinides) are used to establish a setup at this mass. Setups for rarer isotopes are then established by scaling the instrument parameter and subsequent fine tuning using the gas ionization chamber count rate for the isotopes region of interest.

3.5.4 Calibration

Calibration standards are used to determine the instrument sensitivity. These are typically purchased from National Measurement Institutes to ensure traceability and then diluted over a suitable range depending on the instrument capability and the radionuclide activity concentration expected. If instruments with multiple detectors or detector modes are being used, cross-calibration must also be considered. The concentration of the radionuclide of interest should fall as close to the centre of the calibration range as possible.

3.5.4.1 ICP-MS

ICP-MS calibration is typically achieved using isotope dilution (e.g. using ^{242}Pu for Pu isotopes) or external standardisation methods. For MC-ICP-MS, electronic calibrations like gain calibration, dark noise etc. must be performed regularly, ideally before each longer measurement campaign. Mass bias correction must be performed by applying the reference sample bracketing approach.

3.5.4.2 AMS

AMS measures relative isotope ratios and thus needs to use isotopic calibration standards or reference materials for an absolute result in ratio. Typically, after tuning the instrument it is verified that the ratio of the reference material used for normalisation is within the range of expected values based on understood general size of mass bias effects. If the raw isotope ratio of the reference material is not reproduced reasonably it may point to a need for further tuning or underlying technical problem.

3.5.5 Online interference removal

Several mass spectrometry setups have built-in capabilities to support or even replace offline chemical separation. This offers a rapid alternative to offline separation but the balance between interference removal and analyte sensitivity must be considered.

3.5.5.1 ICP-MS

ICP-MS has several approaches to reduce interferences:

- Sample introduction – for example, as described previously, some desolvating sample introduction systems can reduce solvent loading into the plasma, reducing polyatomic interferences from hydrides and oxides. This is beneficial to several radionuclides, including ^{239}Pu through the reduction of ^{238}U^{1}H formation. These systems often have a higher sensitivity and can use low sample volumes compared to 'standard' sample introduction systems and there may be no compromise between sensitivity and interference removal.

- Collision/reaction cell – some instruments are equipped with a pressurised collision/reaction cell prior to the mass analyser. One or more gases can be introduced into the cell to reduce the impact of

interferences through selective reactions with the radionuclide of interest or the interference. Collision gases (such as He) can minimise the impact of polyatomic interferences through collision-based energy reduction, whilst single or mixed reactive gases (such as O_2, NH_3 and N_2O) can react with either the analyte or the isobaric interference, shifting them to an interference-free mass. A range of cell gases have been effectively used (Diez-Fernandez et al. 2024, Lancaster et al. 2025). The reduction in interference must be balanced against the loss of analyte sensitivity, which is particularly important for low-level measurements.

- Cold plasma – the difference in first ionisation energy between a radionuclide and its isobaric interference can be exploited. By reducing the forward power in the plasma from approximately 1500 W to approximately 650 W, the sensitivity of the radionuclide is reduced, but the sensitivity of the isobar with a higher ionisation energy is suppressed more. One example where this has effectively been applied is ^{90}Sr (Liu et al. 2024). The reduction in forward power does make the instrument more vulnerable to matrix effects, which means that additional sample preparation may be needed to produce a sample with very low matrix content.

- High resolution – instruments with varied mass resolution capability can be used to reduce interferences. Operating at a higher mass resolution focuses the central part of the ion beam. Ion transmission is generally reduced as the resolution increases, but interference levels are reduced to a greater extent. Multiple polyatomic interferences can be reduced by operating at higher mass resolution. Unfortunately, for radionuclides measured by mass spectrometry, the resolving power needed to separate isobaric interferences is higher than any commercial instrument can achieve.

3.5.5.2 AMS

AMS systems are naturally suited to destroy molecular interferences, as molecules will be broken up in the gas stripper of the tandem accelerator system and rejected by the high-energy side mass spectrometer. AMS also has several techniques for the removal of isobaric interference:

- Elimination or suppression by negative ion formation (e.g. ^{129}I, where ^{129}Xe does not form negative ions) in the ion source.

- Discrimination in a multi-anode gas ionization detector by exploiting the difference (Z-dependency) of energy loss of the radionuclide versus the interference. This method requires relatively high particle energy (~1MeV/nucleon) to be most effective, however, boosts in isobar suppression can be achieved even at energies below.

- Elimination of interference by using the energy loss difference after a foil followed by a highly resolving energy spectrometer. This method has the advantage that it can handle interference that would overwhelm an ion counter with too high count rates.

- Elimination of interference using a gas-filled magnet detection system. This also needs high particle energies.

- Elimination of interference in an ion-cooler by laser-photo detachment or interaction with a chosen buffer gas: This technique is utilized prior to injection into the tandem accelerator and has already shown drastic isobar suppression for a number of rare isotope-interference pairs otherwise inaccessible to mass spectrometry. For instance, this is now the best method for detecting ^{90}Sr at highest sensitivity.

3.5.6 Limit of detection

The limit of detection achievable depends on factors including the amount of sample, the chemical yield following sample preparation, and instrument parameters such as the mass spectrometer design and instrument background. The limit of detection is typically calculated as 3 times of the standard deviation of the procedure blank.

MC-ICP-MS using IDMS can be used to determine major (%) to ultra-trace concentrations (< pg/g) of elements.

With presence of a stable reference isotope in the samples of interest in AMS the abundance detection limit for the rare isotope relative to the reference isotope is the relevant quantity. This is usually given as the blank level plus two times the uncertainty of the blank level. In the case of carrier or spike addition this definition of the LOD is obviously useless. Here the detection limit can be inferred from processing blanks calculating back to a hypothetical (or actual if a suitable material is available for a full blank) sample mass/volume comparable to the samples. There is no standard as to the definition of this limit to the blank. Typical approaches are:

- Use the blank level plus two times the uncertainty of the blank level

- Based on the 95 % C.I. of the counting statistics for zero count results.

- 3 or 5 times the blank level.

It should be noted that the detection limit may depend on the practical choice for sample used if the abundance in the rare isotope is rather high. Ionisation detectors have a limited capacity for processing count rate which may require throttling of ion source output or using less sample matrix. In this case the detection limit will not reflect the limitations of the instrument for less concentrated samples.

Depending on the instrument and the radionuclide, mass spectrometry is capable of detection limits on the order of 10^{-18} g/g and potentially lower.

3.6 Sources of uncertainty

Uncertainties must be considered throughout the analytical procedure, including sample preparation (weighing, uncertainty in the tracer's concentration, procedure blank, internal standard) and measurement (counting statistics, matric effect, instrument drifting etc). When processing data, conversion between activity and mass and (if relevant) volume and mass must also be considered.

In case of isotope ratios, the major uncertainty components are the certified isotope ratios from the reference material and the measured ion intensities. The order depends on the ratio itself. In case of IDMS experiments, usually the biggest uncertainty sources are content of the reference/ or spike material, followed by weighing of the sample, the reference and the spike. It must be added that corrections for water content or inhomogeneity of the sample can be major contributors as well. In case of single IDMS, a relative expanded uncertainty of less than 2 % can be achieved and in case of double (or reverse) IDMS less than 1 %, using ICP-MS with multiple detectors. For isotope ratio measurements using single detector ICP-MS, the measurement uncertainty is expected to be higher as the detector must 'hop' between masses.

3.7 Instrument maintenance

Mass spectrometry instruments require maintenance at regular intervals. There are often recommendations provided from the manufacturers on the frequency of this maintenance, which may also vary depending on the instrument applications, such as the sample matrices or radioactivity concentrations measured. Examples include changing the sample introduction tubing, cleaning and replacement of the ionisation source, cleaning of the interface region (i.e. lenses), and replacement of oil used in vacuum pumps. With regards to radioactivity measurement, even for very low-level measurement, it is good practice to monitor the components and cleaning equipment, pump oil etc. after maintenance to ensure these are not contaminated.

3.8 Future developments in radionuclide measurement by mass spectrometry

3.8.1 Instrument design

The increasing application of mass spectrometry for radionuclide measurement is leading to developments in instrumentation that is improving both the sensitivities achievable and the online interference removal capabilities. In some cases, the developments are being led by research institutes, such as combining the analytical flexibility of a ICP source with the sensitivity designs such as multi-reflection time of flight[i]. In other cases, there have been recent developments from instrument manufacturers that will potentially benefit radionuclide measurement, such as combining tandem, collision/reaction cell and multi-collector capabilities in a single instrument (e.g. Nu Instruments, 2025).

3.8.2 Standards and reference materials

There are still several radionuclides where suitable tracers, standards and reference materials are not available. This limits the extent to which measurement of these radionuclides by mass spectrometry can be fully realised. There is a current and increasing need to develop these. In the case of standards and reference materials, there are cases where existing options are likely to contain radionuclides of interest to mass spectrometry but may not have been characterised in this way. For standards, recharacterization for mass spectrometric interferences and proving stability and homogeneity at very low levels may be required. There is also an argument to continue developing new standards and radioactive reference materials focused on mass spectrometric measurement, which the MetroPOEM project has contributed to.

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