Deliverable 2.5

Assessment of feasibility of waste form characterisation methods

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Abstract

Deliverable 2.5 collects the various physical/chemical/radiological parameters to be measured and the methods to perform these measurements for the characterization of radioactive waste. This collection aims to help those who are preparing/planning to produce and manage radioactive waste by providing basic information and offering more detailed sources on the parameters and methods regarding radioactive waste characterization. An excel with the same content in searchable/filterable form has been prepared, and creation of a web-based live database from this collection and expanding/completing missing information will be considered.

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LIST OF ABBREVIATIONS

AAS	Atomic Absorption Spectrometry
ABO	α-benzoinoxime=(2E)-2-hydroxyimino-1,2-di(phenyl)ethanol
AEC	Anion Exchange Chromatography
AMP	Ammonium phosphormolybdate
AMP-PAN	Ammonium molybdophosphate impregnated resin
AMS	Accelerator MS
ART	Algebraic reconstruction technique
AS	Alpha spectrometry
ASGS	Absolute SGS
BGO	Bismuth Germanate ($Bi_4Ge_3O_{12}$) scintillator crystal
BS	Beta spectrometry
CEC	Cation Exchange Chromatography
CFC	Copper ferrocyanide
CMPO	Octylphenyldiisobutylcarbamoylmethyl posphineoxide
CRDS	Cavity Ring Down Spectroscopy
Cupferron	N-nitroso-N-phenyl-hydroxylamine
DA	Destructive Analysis/Assay
DEE	Diethyl ether
DGA resin	Diglyolamide coated resin
DIPEX resin=ACTINIDE resin	Bis(2-ethylhexyl) methanediphosphonic acid coated resin
DIPHONIX resin	Diphosphonic acid covalently bound to polystyrene
DMG	Dimethylglyoxime
DTM	Difficult-To-Measure
ECh	Extraction Chromatography
ECT	Emission Computed Tomography
FEP	Full Energy Peak
FWHM	Resolution: Full Width Half Maximum
GDMS	Glow Discharge MS
GS	Gamma spectrometry
HDEHP	Ethyl-hexyl phosphoric acid
HLW	High Level Waste
HRGS	High Resolution Gamma Spectrometry
IART	Improved ART
ICP-MS	Inductively Coupled Plasma - MS
ICP-OES	Inductively Coupled Plasma - OES
ICP-Q-MS	Quadrupole ICP-MS
IER	Ion Exchange Resin
IGS	Integral Gamma Scanning
LA-ICP-MS	Laser Ablation ICP-MS

LILW	Low and Intermediate Level Waste
LINAC	Linear Accelerator
LLE	Liquid-liquid Extraction, solvent extraction
LLE	liquid-liquid extraction, solvent extraction
LN resin	HDEHP coated resin
LoD	Limit of Detection
LSC	Liquid Scintillation Counting
MCS	Multichannel Scaling
MIBK	Methylisobutyl ketone
MS	Mass Spectrometry
NAA, RNAA	Neutron Activation Analysis, radiochemical NAA
NDA	Non-Destructive Analysis/Assay
NPP	Nuclear Power Plant
OES	Optical Emission Spectrometry
PGNAA	Prompt Gamma radiation induced by Neutron Activation
PMBP	Benzoyl pyrazolone
RCh	Radiological Characterization
RCh	radiological characterization
RIMS	Resonance Ionization MS
RW	Radioactive Waste
SF	Spent Fuel
SF-ICP-MS	Sector Field ICP-MS
SGS	Segmented Gamma Scanning
SNS	Segmented Neutron Scanning
Sr resin	4,4'(5')-di-t-butylcyclohexano-18-crown-6 in octanol coated resin
TBP	Tri-butylphosphate
TCT	Transmission Computed Tomography
TEVA resin	Aliquat 336 loaded resin
TGS	Tomographic Gamma Scanning
TIMS	Thermal Ionization - MS
TK 201 disc	TOA impregnated membrane
TK 202 resin	Polyethyleneglycol coated resin
TOA	Tri-octylamine
TOF-MS	Time-Of-Flight MS
TOPO	Tri-octylphosphine oxide
TRU resin	CMPO and TBP coated resin
TTA	Thenoyl-tri-fluor acetone=1-(2'-thenoyl)-3,3,3-trifluoroacetone
UTEVA resin	Dipentylpentyl phosphonate coated resin
XRF	X-ray fluorescence
XS	X-ray spectrometry

1 Introduction

In principle, waste acceptance criteria are edited for waste streams generated by current waste generation operations, in NPPs, front-end and back-end fuel cycle facilities and research centres. For safe disposal, radioactive waste must be characterized to demonstrate its compliance with WAC, including radiological as well as physico-chemical parameters.

Deliverable 2.5 collects the various physical/chemical/radiological parameters to be measured and the methods to perform these measurements for the characterization of radioactive waste. This collection aims to help those who are preparing/planning to produce and manage radioactive waste by providing basic information and offering more detailed sources on the parameters and methods regarding radioactive waste characterization. An excel with the same content in searchable/filterable form has been prepared, and creation of a a web-based live database from this collection and expanding/completing missing information will be considered.

2 Parameters and Methods

In this section the collected parameters and methods are shown.

2.1 Radiological parameters

2.1.1 Fe-55

Property	Fe-55
Measurement method	LSC X-ray spectrometry
Property info	It is a pure β- decaying isotope, it decays through electron capture to Mn-55 emitting characteristic X-rays of 5.9 keV (24%) and Auger electrons of 5.2 keV (60%). Its half-life is 2.73 y (short-lived). Fe-55 is produced by n capture from Fe-54 (5.8% of stable Fe) with high neutron cross section (σ=2.25 barn). Since iron is a major construction material in nuclear facilities Fe-55 is responsible for a significant part of the induced radioactivity on relatively short term: More than 50% of the total activity accumulated in a nuclear reactor is due to Fe-55 a couple of years after reactor shutdown, thus it is a major contributor to decommissioning waste. Fe-55 can be determined by X-ray spectrometry using low energy Ge or Si detectors or by LSC. Fe-55 is a DTM nuclide, due to the low energy of the emitted radiations radiochemical separation is necessary. Stable Fe presents the most significant limitation in the sensitive determination of Fe-55. It produces an intensive colour in the LSC source resulting in strong quenching and makes X-ray sources thick causing self-absorption losses in X-ray spectrometry. To reduce quench effects, complexing agent such as HF, oxalic acid or a reducing agent such as ascorbic acid are given to the LSC source. Counting efficiency is calculated as a function of quench parameter. The chemical recovery can be determined by measuring stable Fe content of the sample before and after the chemical operations using optical (AAS, ICP-OES) or mass spectrometric techniques (ICP-MS).
Technical info, interferences	• LSC efficiency is calibrated as a function of quench parameter • X-ray counting efficiency is calibrated as a function of source thickness • interferences in LSC: stable Fe due to color, low energy β emitters (H-3, Ni-63, Pu-241...) • interferences in XS: stable Fe due to its mass • In the case of direct measurement of mineralized samples, some nuclides can interfere depending on the nature of sample. Ca-41, Ni-59, Cd-109 and Pu-241 are described as main interferences because their maximum beta energy is in the same region as the one of Fe-55 (~6 keV). • Gamma emitters interference is checked out from the sample to be measured in order to discount its contribution in the sample counting.

Sample preparation	• Fe pre-concentrated usually as hydroxide precipitate using ammonia • LLE: $FeCl_3$ is extracted from 8M HCl with di-isopropyl ether or MIBK; Fe Cupferron complexes are extracted from 2M HCl with $HCCl_3$ • Ion exchange: $FeCl_3$ is retained by cation exchangers from 0.2M HCl; Fe as $[FeCl_4]^-$ is retained on anion exchange resin from concentrated HCl solution, it is eluted with dilute acid. • ECh: Fe(III) nitrate complex is retained by TRU resin from 8M HNO_3 and is stripped with 2M HNO_3; DGA resin can retain FeCl3 from HCl solutions of 4M or higher The single separation steps are usually combined.
Chemical yield evaluation	• measurement of stable Fe by AAS, ICP-OES, ICP-MS before and after separation
MDA/Detection limit	LSC: MDA for analyte is > 0.1 Bq/sample depending on stable Fe content X-ray spectrometry: 1 Bq/sample
Sources - detailed information	• Hou, X. I., Ostergaard, L. F., Nielsen, S. P. (2005): Radiochemical determination of Fe-55 and Ni-63 in nuclear waste samples, Anal. Chim. Acta, 535, 297. • CEA: Determination of iron-55 in water. Method 396, Laboratory Procedures Manuel, CEA/CETAMA Ni-63 and Fe-55 Interlaboratory comparison (ILC), 2019.

2.1.2 Ni isotopes: Ni-59, Ni-63

Property	Ni isotopes: Ni-59, Ni-63
Measurement method	LSC: Ni-63 X-ray spectrometry: Ni-59 AMS: Ni-59
Property info	Ni-59 decays by electron capture to stable Co-59 emitting characteristic X-rays of 6.9 keV (31%) and Auger electrons of 6.1 keV (54%), its half-life is $7.6 \cdot 10^4$ y. Ni-63 is a pure β- emitter, it decays to stable Cu-63. The maximum energy of the β electrons is 67 keV, its half-life is 100.1 y. Both Ni isotopes are produced from stable Ni by neutron capture in the Ni-58 (n,γ) Ni-59 and Ni-62 (n,γ) Ni-63 reactions, respectively, with high cross sections (σNi-59=4.6 b, σNi-63=14.2 b). Ni-58 (68% abundance) and Ni-62 (3.6% abundance in Ni) are important components of the steel construction materials and therefore Ni-59 and especially Ni-63 contribute significantly to the accumulated activity in nuclear reactors. The activity of Ni-63 is comparable with that of Co-60 at the end of operation and a couple of years after reactor shutdown and its relative activity increases with longer cooling time. The typical activity ratio of Ni-63/Ni-59 is around 100. Ni-59 can be detected by X-ray spectrometry and Ni-63 by LSC. Because the activity of Ni-63 is much higher than that of Ni-59, and the LS spectra of Ni-59 (X-ray and Auger electron spectrum) and Ni-63 (β spectrum) are overlapping therefore only Ni-63 is measurable by LSC. For high sensitivity measurements of Ni-59 AMS can be used. Both isotopes are DTM nuclides. Ni has to be separated from the matrix, since any other radionuclides present would interfere in LSC or X-ray detection. For X-ray spectrometry thin sources have to be prepared to reduce self-absorption. If Ni-59 is detected by AMS, stable Co has to be completely removed.
Technical info, interferences	• LSC efficiency is calibrated as a function of quench parameter • X-ray counting efficiency is calibrated as a function of source thickness • interferences in LSC: low energy β emitters (H-3, Ni-63, Pu-241...), Ni-59 • interferences in XS: stable Co • interferences in AMS: isobaric Co-59
Sample preparation	• Ni (and Fe) can be pre-concentrated usually as hydroxide precipitate • After removal of Fe, Ni is precipitated in the presence of ammonium citrate with DMG at pH 8-9, the precipitate is dissolved with 8M HCl • After removal of Fe, Ni is retained (precipitated) on Ni resin containing DMG in the presence ammonium citrate at pH 8-9 , Ni-DMG_2 precipitate is eluted with 3M HNO_3. • For AMS measurement of Ni-59, Ni is separated as volatile Ni tetracarbonyl $Ni(CO)_4$.
Chemical yield evaluation	• measurement of stable Ni by AAS, ICP-OES, ICP-MS before and after separation
MDA/Detection limit	LSC: MDA for analyte (Ni-63) is >0.1 Bq/sample depending on the quench parameter X-ray spectrometry: 1 Bq/sample AMS (Ni-59)?

Sources - detailed information	• Hou, X. I., Ostergaard, L. F., Nielsen, S. P. (2005): Radiochemical determination of Fe-55 and Ni-63 in nuclear waste samples, Anal. Chim. Acta, 535, 297. • TRISKEM-International Co.: NI resin, https://www.triskem-international.com/catalog/ products/resins-and-accessories/ni-resin/bl,product,408,0 • CEA: Determination of nickel-63 in effluent. Method 398, Laboratory Procedures Manuel, CEA/CETAMA Ni-63 and Fe-55 Interlaboratory comparison (ILC), 2019. • Céline Gautier, Christèle Colin, Cécile Garcia, (2016) A comparative study using liquid scintillation counting to determine Ni-63 in low and intermediate level radioactive waste, J Radioanal Nucl Chem 308:261–270 • McAninch, J. E., Hainsworth, L. J., Marchetti, A. A., Leivers, M. R., Jones, P. R., Dunlop, A. E., Mauthe, R., Vogel, S., Proctor, I. D., Straume, T. (1997), Measurement of Ni-63 and Ni-59 by accelerator mass spectrometry using characteristic projectile X-rays. Nucl. Instrum. Methods B, 123, 137 • Developing AMS measurement of Ni-59 at CIAE - He Ming, Wang Wei, Ruan Xiangdong, Li Chaoli, Dong Kejun, Du liang, Xie Lin Bo, Li Zhenyu, Zhen Guowen, Hu Hao, Liu J

2.1.3 Zr-93

Property	Zr-93
Measurement method	LSC ICP-MS
Property info	Zr-93 is a pure β- decaying radionuclide emitting low energy β- electrons (β_{Max} 60 keV), its half-life is long ($T_{1/2}=1.5*10^{6}$ y). It is produced by neutron capture from stable Zr-92(n, γ) where the isotope abundance of Zr-92 is 17.1% and the reaction cross section is moderately high (σ=0.26 b). The cladding material of the fuel elements in the thermal reactors operated world-wide is Zr alloy, and soil and concrete also contain some Zr. Due to its very long half-life Zr-93 is a radiologically important radionuclide on long-term in nuclear facilities and radioactive wastes. Zr-93 is a DTM nuclide that can be detected by ICP-MS and LSC. ICP-MS is theoretically more sensitive for Zr-93 but the presence of stable Zr in the samples reduces the sensitivity because of the abundance sensitivity due to the presence of stable Zr-92 and Zr-94 nuclides and isobaric interferences first of all due to Nb-93 (isotope abundance of Nb-93 is 100%) and the radioactive Mo-93 ($T_{1/2}=4*10^{3}$ y) that is also produced from stable Mo in steel samples in nuclear reactors. When measuring by LSC, complete separation from the matrix and pure low energy β- and X-ray/Auger electron emitting radionuclides (e.g., H-3, C-14, Fe-55, Ni-63, Mo-93, Pu-241) is necessary. One of the major issues in Zr chemistry is the strong affinity to hydrolyse and form polymers. Hydrolysis can occur in 1M acid solutions. Increasing the pH, various hydroxide complexes appear and the very low solubility $Zr(OH)_4$ precipitate forms. Zr oxides are soluble in HF. Zr can be pre-concentrated as ZrO_2*nH_2O or as $BaZrF_6$ precipitates.
Technical info, interferences	• interferences in LSC: low energy β emitters (H-3, C-14, Fe-55, Ni-63, Mo-93, Pu-241...) • interferences in ICP-MS: isobaric: stable Nb (Nb-93 100%), Mo-93, abundance sensitivity: stable Zr (Zr-92, Zr-94)
Sample preparation	• Zr is pre-concentrated as ZrO_2*nH_2O or $BaZrF_6$ precipitates. • Zr is extracted from 2 M HNO_3 using 0.5 M TTA and back-extracted with 2 M HF. • Zr is extracted from 4 M HCl/1M $AlCl_3$ using 0.5 M TTA and back-extracted with 0.25 M HNO_3/HF. • Zr is extracted with ABO (α-benzoinoxime). • Zr forms a complex with 5% Cupferron that is extracted in to $HCCl_3$ and back-extracted with 6M HNO_3. • Zr is retained from 7 M HNO_3 on TBP resin and stripped with 0.3 M HNO_3. • Zr, Nb and Mo are retained on TEVA resin from 9 M HCl. Zr is eluted with H_2O. • Zr is retained on TEVA from >9M HCl, Zr is stripped with 8M HF/0.01M HF. • Zr, Nb, Hf and Ta are retained on UTEVA resin from 8 M HNO_3 or from 9M HCl, Zr is stripped with 4M HCl. • Zr is retained on TRU resin from 8-10M HNO_3. • Zr is retained on DGA resin from 4M HCl and stripped with 0.5M HNO3/0.5% oxalic acid.

	• Zr is retained on Zr resin (containing hydroxamate) from HCl and HNO_3 of a wide range of acidity.
Chemical yield evaluation	• measurement of stable Zr by AAS, ICP-OES, ICP-MS before and after separation • measurement of Zr-95 tracer by γ spectrometry
MDA/Detection limit	LSC: 100 mBq/sample ICP-MS: 0.01 Bq/sample
Sources - detailed information	• Espartero, A. G., Suarez, J. A., Rodrigez, M., Pina, G. (2002) Radiochemical analysis of Zr-93. Appl. Radiat. Isot., 56, 41. • Chartier, F., Isnard, H., Degros, J. P., Faurce, A. L., Frechou, C. (2008): Application of the isotope dilution technique for Zr-93 determination in an irradiated cladding material by multiple collector ICP-MS. J. Mass Spectrom., 270, 133. • TRISKEM-International Co: TBP resin, https://www.triskem-international.com/ scripts/files/5f463448ae8852.59615909/PS_TBP-Resin_EN_160927.pdf • Development of HCl-free solid-phase extraction combined with ICP-MS/MS for rapid assessment of difficult-to-measure radionuclides. Part I. Selective measurement of Zr-93 and Mo-93 in concrete rubble Van-Khoai Do; Takahiro Furuse; Erina Murakami; Rena Aita; Yuki Ohta; Soichi Sato • Horwitz, E. P., Dietz, M., Chiarizia, R., Diamond, H., Maxwell, S. L., Nelson, M. R. (1995). Separation and preconcentration of actinides by extraction chromatography using a supported liquid anion exchanger: application to the characterization of high-level nuclear waste solutions. Anal. Chim. Acta 310, 63-78. • Horwitz, E. P. (1992). Separation and preconcentration of uranium from acidic media by extraction chromatography. Anal. Chim. Acta 266, 25-37. • Sz. Osváth, N. Vajda, Zs. Molnár, É. Széles, Zs. Stefánka: Determination of Np-237, Zr-93 and other long-lived radionuclides in medium and low-level radioactive waste samples. Journal of Radioanalytical and Nuclear Chemistry, 286, 3 (2010) 675-680 • Horwitz P., Chiarizia R. Dietz M., Diamond H., Nelson, D.; Separation and preconcentration of actinides from acidic media by extraction chromatography. Analytica Chimica Acta, 281, pp. 361-372 (1993) • Horwitz, E. P. , McAlister, D. R., Bond, A. H., and Barrans, R. E. Jr. (2005). Novel extraction chromatographic resins based on tetraalkyldiglycolamides: Characterization and potential applications. Solvent Extr. Ion Exchange 23, 319-344. • TRISKEM-International Co: ZR resin, https://www.triskem-international.com/ scripts/files/61eef0f5e9fa69.45400285/PS_ZR-Resin_EN_210908.pdf

2.1.4 Nb isotopes: Nb-93m, Nb-94

Property	Nb isotopes: Nb-93m, Nb-94
Measurement method	Gamma Spectrometry: Nb-94 ICP-MS: Nb-94 X-ray spectrometry: Nb-93m
Property info	Nb-94 is a β- decaying radionuclide emitting medium energy β- electrons (β_{Max} 472 keV) and easy to measure γ radiation at 703 keV (98%) and 871 keV (100%), its half-life is long ($T_{1/2}$=2.03*10^4 y). It is produced by neutron capture from stable Nb: Nb-93(n, γ) where the isotope abundance of Nb-93 is 100% and the reaction cross section is moderately high (σ=1.15 b). Nb-93m decays by isomer transition to stable Nb-93 that emits by internal conversion X-rays at 16.6 keV (about 10%), its half-life is in the short range ($T_{1/2}$=16.1 y). It is also produced by neutron capture from stable Nb: Nb-93(n, n) where the isotope abundance of Nb-93 is 100%. Nb-93 is the decay product of both Zr-93 and Mo-93. Stable Nb is a structural component of the Zr alloys used as cladding material in most of the nuclear reactors. Therefore, both Nb-93m and especially Nb-94 are radiologically important components in radioactive wastes. Nb-94 is not strictly a DTM nuclide but due to its relatively small specific activity radiochemical separation is needed for the radiometric determination using γ spectrometry. Nb-93m can be measured by X-ray spectrometry while Nb-94 is measured by γ spectrometry or LSC or ICP-MS. Nb-93m cannot be detected by LSC in the presence of Nb-94.
Technical info, interferences	• interferences in γ spectrometry: high radioactivity elevating the Compton background • interferences in ICP-MS of Nb-94: isobaric stable Mo-94: stable abundance sensitivity: stable Mo (Mo-95), stable Nb-93 • interferences in X-ray spectrometry: matrix via source thickness
Sample preparation	• Nb is pre-concentrated as Nb_2O_5 precipitate. • Nb is extracted from 4M HCl/1M $AlCl_3$ using ABO and back-extracted with 4M NH_4OH. • Zr, Nb, Sb are retained on anion exchange resin from 7M HF (or > 7M HF), Zr is stripped with 6M HCl, Nb is stripped with 2M HBr/0.5M HF, Sb remains on resin • Zr, Nb, Mo are retained on TEVA resin from 11M HCl, Zr is stripped with 8M HCl/0.01M HF, Mo with 4M HCl and Nb with 7M HNO_3
Chemical yield evaluation	• measurement of stable Nb by AAS, ICP-OES, ICP-MS before and after separation • measurement of Nb-95 tracer by γ spectrometry
MDA/Detection limit	γ spectrometry: 1 Bq/sample ICP-MS: 0.01 Bq/sample X-ray spectrometry: ?
Sources - detailed information	• Bombard, A.: Determination of the long-lived radionuclides Zr-93, Mo-93 and Nb-94 in nuclear waste. PhD thesis, Universite de Nantes, 2005. • Osváth, Sz., Vajda, N., Molnár, Zs., (2008) Determination of long-lived Nb isotopes in nuclear power plant wastes. Applied Radiat. Isot., 66, 24. • IAEA-TRS No. 389: Radiochemical Characterization of Shut Down Nuclear Reactors for Decommissioning Purposes, 1998

2.1.5 Sr-90 (Sr-89)

Property	Sr-90 (Sr-89)
Measurement method	Gross beta counting (proportional counter via Y-90) LSC Cerenkov counting TIMS AMS
Property info	It is a pure β- emitting isotope of maximum β energy in the medium energy range (β_{Max} 546 keV). Its short-lived decay product is Y-90 that gets in secular equilibrium with the parent in a couple of weeks. Y-90 is also a pure β- emitting isotope, of maximum β energy in the high energy range (β_{Max} 2270 keV). Due to its high fission yield, short-medium half-life (28 y), high energy β radiation of its daughter, and its bone seeking nature Sr-90 is a radiologically very important fission product on medium time scale for waste management. Sr is a DTM isotope, for its determination by LSC, other β counting techniques, or recently by means of MS (TIMS, AMS) radiochemical separation is required. When β counting techniques are used, the sample matrix has to be removed including all other alkaline earth metals. Till recent times the major issue was the separation of Sr from the typically much greater concentration of Ca, but this difficulty has been overcome by the use of Sr specific crown ether extractant (Sr resin). Sr-89, a pure β- emitting isotope of 50 d half-life might also cause interference if samples are not “old”. By repeated β counting the interference can be eliminated. If Sr-90 is detected by ICP-MS, the major interference in the Sr source is stable Zr-90, chemical separation has to assure the efficient removal of the matrix to get low salt content in the measuring source. For the selective separation of Sr several procedures have been developed and are in use in waste characterization. The conventional method is based on a series of precipitations: pre-concentration of alkaline earth metals as carbonate or oxalate precipitates, purification with Fe hydroxide scavenge, separation of Sr and Ca as nitrate precipitate with or without the use of fuming nitric acid, separation of Sr and Ba as chromate precipitate. This method is still useful when large amounts of Sr have to be separated, e.g., when more than 100 L of sea water is analysed for Sr-90. Later certain steps of the procedure were replaced by more efficient chromatographic separation. Cation exchange resins were used in the presence of specific complexing agents (e.g., EDTA) for the separation of Sr, but cation exchangers are not highly selective for Sr against Ca and Ba.

Technical info, interferences	Beta counting (proportional counting): • The efficiency is calculated with Sr-89 and Sr-90/Y-90 certified reference solutions as a function of source thickness. • By repeated counting both Sr-90 and Sr-89 can be determined. • No special interferences are described LSC: • The LSC efficiency is calculated with Sr-89 and Sr-90/Y-90 certified reference solutions. The efficiency obtained is practically 100% at the typical quenching values. The window setting is 0-2000 keV. • Two or three window counting techniques are possible. • By repeated counting both Sr-90 and Sr-89 can be determined. Cerenkov: • Sr-89 and Y-90 can be counted by detection of Cerenkov radiation using LSC instrument. • No special interferences are described AMS/TIMS: '• Only Sr-90 can be determined. • Possible interference: stable Zr-90
Sample preparation	• Selective separation of Sr by a series of precipitations (nitrate, chromate, carbonate, oxalate) • Separation of Sr by cation exchange chromatography (CEC) in presence of specific complexing agent (EDTA) • Liquid-liquid extraction of Y with TBP or HDEHP and back-extraction with dilute acid • Pre-concentration of Sr with precipitation followed by its selective separation by ECh using crown ether based Sr resin or a combination of the procedures with additional purifications • Y can be selectively separated by ECh with diglycolamide based DGA resin • Preparation of Sr and/or Y source by precipitation as oxalate or carbonate. AMS/TIMS: The same as for LSC, but the final Sr source is liquid, e.g. the eluate of the Sr resin.
Chemical yield evaluation	• gravimetric yield determination of a Sr precipitate, e.g., Sr oxalate or carbonate • measurement of stable Sr by AAS, ICP-OES, ICP-MS • measurement of Sr-85 tracer by γ spectrometry or by direct LSC
MDA/Detection limit	Beta counting: As low as ~0.03 Bq/kg LSC: MDA for analyte is 10-100 mBq/sample depending on the activity of other Sr, Y nuclides TIMS: Sr-90: < 0.1 Bq/sample AMS: > 5 Bq

Sources - detailed information	• Horwitz, E. P., Dietz, M. L., Fisher, D. E., 1991b. Separation and Preconcentration of Strontium from Biological, Environmental, and Nuclear Waste Samples by Extraction Chromatography Using a Crown Ether. Anal. Chem. 63, 522-525. • Groska, J., Molnar Z, Bokori E, Vajda N (2012) Simultaneous determination of Sr-89 and Sr-90: comparison of methods and calculation techniques J Radioanal Nucl Chem 291:(3) pp. 707-715. • Vajda, N., Kim, C. K., Determination of radiostrontium isotopes: A review of analytical methodology, Appl. Radiat. Isot. 68 (2010) 2306-2326).

2.1.6 Cs-135

Property	Cs-135
Measurement method	NAA ICP-MS
Property info	It is a pure β- emitting isotope of maximum β energy in the medium energy range (β_{Max} 270 keV). Its short lived (29 h) daughter Ba-135m emits γ line at 268 keV (16%). Due to its high fission yield (from U-235 6.5%), very long half-life (2.3*106 y) and the high mobility it is a radiologically important fission product on long term for radioactive waste and the environment of nuclear facilities. Cs-135 is a DTM isotope, for its sensitive determination neutron activation analysis (NAA) and ICP-MS can be applied. The γ spectrometric measurement via its Ba-135m daughter is usually not sensitive because samples containing Cs-135 also contain Cs-137 and most likely Cs-134 of much higher activity than that of Cs-135. On the high Compton background the 268 keV line is not detectable. When Cs-135 is irradiated by neutrons the short lived ($T_{1/2}$=13 d) Cs-136 is produced that emits easy to measure γ radiation at 818, 1048, 1235 keV. The major interference in this method comes from the radioactivity of Cs-137 and Cs-134. The latter one is produced as a result of neutron irradiation of stable Cs, besides Cs-134 present in the sample originally. Cs-135 can be determined by ICP-MS. Main interferences originate from abundance sensitivity due to stable Cs-133, and isobaric nuclide Ba-135. Spectrometric corrections for both interferences are possible but the accuracy of the results will be reduced. MS measurements by TIMS and especially RIMS have higher sensitivity than ICP-MS.
Technical info, interferences	• NAA: Cs-136 is measured by γ spectrometry at 818, 1048, 1235 keV, interference in NAA: Cs-134, Cs-137 • ICP-MS: interference: isobar Ba-135
Sample preparation	• AMP retains Cs by ion exchange from 0.1M HCl, the precipitate is dissolved with 10M NaOH. Copper ferrocyanide (CFC), potassium cobalt hexacyanoferrate, zirconium phosphate can retain Cs • Cation exchange chromatography is used to purify Cs: sample is loaded from 2% EDTA-0.75M NaOH, Ba is removed with EDTA, alkali metals are eluted with dilute HCl, Cs is eluted with 3M HCl. • AMP-PAN resin retain Cs isotopes from less than 1M HNO_3 and can be eluted using 1.5 M $NH_3 \cdot H_2O$
Chemical yield evaluation	• measurement of Cs-137 by γ spectrometry • Stable Cs by MS
MDA/Detection limit	NAA: 10-50 mBq/sample ICP-MS: 10-50 mBq/sample

Sources - detailed information	• Nagy P, Vajda N, Sziklai-László I, Kovács-Széles É, Simonits A (2014). Determination of Cs-135 in nuclear power plant wastes by ICP-MS and k0-NAA. J. Radioanal. Nuclear Chem. 299:(2) pp. 615-627. (2014) • Zhu, L., Hou, X., Qiao, J. (2021) Determination of Cs-135 concentration and Cs-135/Cs-137 ratio in waste samples from nuclear decommissioning by chemical separation and ICP-MS/MS, Talanta 221:121637. • Horwitz, E. P. ; Chiarizia, R., Dietz, M. L. (1997) DIPEX: A new extraction chromatographic material for the separation and preconcentration of actinides from aqueous solution, Reactive Functional Polymers 33, 25-36.

2.1.7 Sb-125

Property	Sb-125
Measurement method	Gamma Spectrometry
Property info	It is a short-lived ($T_{1/2}$=2.76 y) β- decaying radionuclide emitting high energy β- electrons (β_{Max} 2350 keV) and easy to measure γ radiation at 428 keV (30%) and 601 keV (18%). It is produced from Sn-124 in the Sn-124(n,γ) Sn-125 reaction (target abundance 5.8 %, cross section σ=0.13 b) and consecutive β- decay. Tin is an alloying metal of Zircaloy (0.25-1.7%) the main cladding material of nuclear reactors in western countries. It is also produced by fission but the fission yield is low (0.03% for U-235). It is basically an Easy to Measure (ETM) nuclide that can be measured by high resolution γ spectrometry, but to increase the detection sensitivity it has to be radiochemically concentrated.
Technical info, interferences	• The 428 keV (30%), 601 keV (18%) etc. γ lines are measured by γ spectrometry together with the 603 keV (98%), 1691 keV (50%) γ lines of Sb-124 tracer.
Sample preparation	• Antimony forms insoluble Sb2S3 precipitate that can be used pre-concentration • The volatile hydride of Sb (with As, Se and Te) is produced with sodium borohydride • Sb (with Zr and Nb) is retained on anion exchange resin from 6M HF. Zr is eluted with 22M HF, Nb with 2M HBr/0.5M HF. Sb remains on the resin
Chemical yield evaluation	• measurement of Sb-124 tracer by γ spectrometry • measurement of stable Sb by AAS, ICP-OES, ICP-MS
MDA/Detection limit	γ spectrometry: 1 Bq/sample
Sources - detailed information	• Osváth, Sz., Molnár, Zs., Groska, J., Vajda, N. (2016) Determination of 125Sb in nuclear power plant wastes. J. Radioanal. Nuclear Chem. 307:761–764

2.1.8 Pu isotopes: Pu-238, Pu-239, Pu-240, Pu-241

Property	Pu isotopes: Pu-238, Pu-239, Pu-240, Pu-241
Measurement method	Alpha Spectrometry: Pu-238, Pu-239,240 ICP-MS: Pu-239, Pu-240 LSC: Pu-241
Property info	Long-lived Pu nuclides – with the exception of Pu-241 - are α emitters. The main α lines of Pu-239 and Pu-240 cannot be resolved with standard α spectrometers where the FWHM is 20-40 keV, the peaks can only be resolved if high resolution α spectrometer and appropriate spectrum fitting software are applied. The α peak of Pu-238 is generally well separated from those of Pu-239/240. Pu-241 is β- decaying isotope emitting low energy β electrons (β_{Max} 20.8 keV). Its long-lived decay product is Am-241. Due to the long half-lives, emission of highly ionizing α particles, and the bone seeking nature Pu nuclides are radiologically very important on medium-long time scale for waste management. Plutonium isotopes are all DTM nuclides, for the α emitters α spectrometry is the standard measuring technique. From the α spectra the activities of Pu-238 and Pu-239/240 can be calculated. Because of the long and similar half-lives of the latter ones (2.41*104 y and 6.56*103 y) the usual practice is to determine their gross activities. Activities of Pu-239 and Pu-240 can be determined separately by ICP-MS. The detection limits in case of α spectrometry and ICP-MS are comparable. Unfortunately, Pu-238 cannot be determined accurately by ICP-MS due to the isobaric interference from U-238 that usually has significantly higher activity concentration than Pu-238 in waste samples. Recently other mass spectrometric techniques (TIMS, AMS, RIMS) have also been used. For the detection of the low energy β particles emitted by Pu-241 LSC is the standard counting technique.
Technical info, interferences	• The α peaks of Pu-239 and Pu-240 cannot be resolved by standard Si α spectrometry • Pu-239 and Pu-240 can be resolved by ICP-MS, but the sensitivity for Pu-238 is poor. High DF for U-238 is desirable. • Pu-241 can be selectively measured by LSC because α peaks of other Pu isotopes appear at higher energy region in LSC spectrum. α/β separation option in LSC is useful.

Sample preparation	• Pu nuclides (together with other actinides) can be pre-concentrated by co-precipitation with Fe (ferrous or ferric) hydroxide using Fe as a carrier, with Mn dioxide, Ca phosphate, lanthanide fluorides. Resins of very high distribution ratio (Diphonix resin, Dipex resin) can also be used for actinide pre-concentration. • Separation of Pu(IV) by anion exchange chromatography (AEC) can be done from HNO_3 or HCl. The oxidation state of Pu(IV) is adjusted with $NaNO_2$ or H_2O_2.The eluate of HNO_3 8M is used for Am/Cm determination. The retained plutonium is stripped as Pu(III) with HCl containing a reducing agent (NH_4I or $TiCl_3$). • Separation of Pu by liquid-liquid extraction (LLE) using ion association complexes such as t-butyl phosphate (TBP), t-octylphosphine oxide (TOPO), t-octylamine (TOA), using chelate extractants such as ethylhexyl-phosphoric acid (HDEHP), thenoyltrifluoroacetone (TTA) after oxidation state adjustment. • After pre-concentration and adjustment of the valence states of actinides Pu(IV) is separated by AEC as written above or by ECh using one of the actinide specific ECh resins: TEVA resin, UTEVA resin, TRU resin, DGA resin. • Pu sources are prepared by electroplating or micro-co-precipitation for α spectrometry. Pu strip solution optionally after further purification is used for ICP-MS. LSC sources are prepared by mixing the strip solution with LS cocktail, or by dissolution the microprecipitated α source and mixing it with LS cocktail.
Chemical yield evaluation	• In α spectrometry samples are spiked with Pu-236 or Pu-242 certified reference solutions. The tracer activity is measured in the source by α spectrometry. • In ICP-MS Pu-242 tracer is used for chemical yield determination • Chemical yield can be estimated from the difference of gross α activities after and before extraction of Pu/U. (Am-241 activity is determined by gamma−spectrometry and subtracted from the gross alpha determination before extraction).
MDA/Detection limit	α spectrometry: 0.1 mBq/sample SF-ICP-MS: 1 pg in 1 mL; Pu-239, Pu-240: < 0.05 mBq/sample AMS: Pu-239, Pu-240: < 0.002 mBq/sample TIMS: Pu-239: 0.002 mBq/sample (1 fg) LSC: Pu-241: 10 mBq/sample

Sources - detailed information	• Horwitz, E. P., Dietz, M., Chiarizia, R., Diamond, H., Maxwell, S. L., Nelson, M. R. (1995). Separation and preconcentration of actinides by extraction chromatography using a supported liquid anion exchanger: application to the characterization of high-level nuclear waste solutions. Anal. Chim. Acta 310, 63-78. • Horwitz, E. P. (1992). Separation and preconcentration of uranium from acidic media by extraction chromatography. Anal. Chim. Acta 266, 25-37. • Horwitz P., Chiarizia R. Dietz M., Diamond H., Nelson, D.; Separation and preconcentration of actinides from acidic media by extraction chromatography. Analytica Chimica Acta, 281, pp. 361-372 (1993) • Horwitz, E. P. , McAlister, D. R., Bond, A. H., and Barrans, R. E. Jr. (2005). Novel extraction chromatographic resins based on tetraalkyldiglycolamides: Characterization and potential applications. Solvent Extr. Ion Exchange 23, 319-344. • Horwitz, E. P. ; Chiarizia, R., Dietz, M. L. (1997) DIPEX: A new extraction chromatographic material for the separation and preconcentration of actinides from aqueous solution, Reactive Functional Polymers 33, 25-36. • TRISKEM-International Co: Actinide resin, https://www.triskem-international.com/ scripts/files/5f46337772d5f9.20794540/PS_AC-Resin_EN_150909.pdf • Vajda, N., Kim, C. K., Determination of plutonium isotopes by α spectrometry: A review of analytical methodology, J. Radioanal. Nucl. Chem. 2010, 283, 203-223 • IAEA-TRS No. 389: Radiochemical Characterization of Shut Down Nuclear Reactors for Decommissioning Purposes, 1998

2.1.9 Am, Cm isotopes: Am-241, Am-243, Cm-242, Cm-243, Cm-244

Property	Am, Cm isotopes: Am-241, Am-243, Cm-242, Cm-243, Cm-244
Measurement method	Gamma Spectrometry: Am-241, Am-243 Alpha Spectrometry: Am-241, Am-243, Cm-242, Cm-243, Cm-244 ICP-MS: Am-241
Property info	The long-lived Am and Cm nuclides, i.e., Am-241, Am-243, Cm-242, Cm-243, Cm-244 belong to minor actinides according to the terminology in nuclear industry. They are all α emitters. The half-life of Cm-242 is 163 d therefore it is not relevant for characterization of “old” radioactive waste. The most sensitive technique for their determination is α spectrometry. The radiologically most important nuclide is Am-241 ($T_{1/2}$=432 y) that is formed by the β decay of the parent Pu-241. Its main α line (5486 keV) overlaps with that of Pu-238 (5499 keV) therefore a good separation of Am from Pu is necessary. The production rate of Am-243 in the actinides transmutation is much smaller than that of Am-241, therefore it is often used as a radionuclide tracer for Am, Cm determination. The α energies of Am-241 (5486 keV) and Am-243 (5275 keV) are relatively close to each other, for this reason also a good quality (thin) α source is needed, and an Am-lanthanide separation is often required to remove lanthanides and to avoid that sources become thick. The α energies of Cm-243 (5785 keV) and Cm-244 (5805 keV) are close to each other, they cannot be resolved by standard α spectrometry, high resolution α spectrometry provides the best chance for their spectrometric resolution. Due to their comparable half-lives (29 and 18 y, respectively) and the much lower production rate of Cm-243, their gross activities are usually calculated or the activity of Cm-243 is simply neglected. Am-241 and Am-243 can be measured by γ spectrometry at the 59 keV and 75 keV γ lines, respectively with low energy γ spectrometer, but the sensitivity is much lower than that in α spectrometry. To increase the sensitivity chemical concentration/separation is desirable also for γ spectrometry. Am-241 and Am-243 can be measured by ICP-MS; the major interference is Pu-241. Am-241 can be detected by other powerful MS techniques, e.g., TIMS, AMS, RIMS, but these techniques cannot be regarded as routine ones in waste characterization. A review about the mass spectrometric determination of Am-241 has been prepared by S. K. Aggarwal. Americium and Cm nuclides are all DTM nuclides, for their determination radiochemical separations are needed. Am and Cm due to their chemical similarities are usually processed together. Both form trivalent actinide species in most of the aqueous solutions. Am(III) can be oxidized with very strong oxidizing agent such as $K_2S_2O_8$ together with Ag+ catalyst to Am(V) while Cm remains still trivalent but traces of reducing agent will reverse the reaction. The trivalent species are similar to lanthanides (Ln’s) therefore separation of Am, Cm from Ln’s is a challenging job that is unavoidable if samples like soils contain Ln’s in mg amounts or more.

Technical info, interferences	• Am-241 and Am-243 can be measured by γ spectrometry at the 59 keV and 75 keV γ lines, resp. with low energy γ spectrometer • the activity of Am-243 is typically orders of magnitude smaller than that of Am-241, therefore Am-243 can be used as yield tracer in the analysis • all Am and Cm nuclides are α emitters and can be measured by α spectrometry; the α energies of Cm-243 (5785 keV) and Cm-244 (5805 keV) are close to each other, they cannot be resolved by standard α spectrometry, high resolution α spectrometry is required; a good quality (thin) source is needed to resolve the α energies of Am-241 (5486 keV) and Am-243 (5275 keV), for that purpose an Am-lanthanide separation is often necessary; Pu-238 can interfere with Am-241 determination in α spectrometry • Am-241 and Am-243 can be measured by ICP-MS; the major interference is Pu-241 • Am-241 can be detected by other MS techniques, e.g. TIMS, AMS, RIMS)
Sample preparation	•Am, Cm can be pre-concentrated with Fe hydroxides, Ca oxalate, Ca fluoride, lanthanide fluorides and hydroxides, MnO_2, $BaSO_4$, Ca phosphate, Ca/Mg carbonate and by ECh with Diphonix and Dipex resins • Am, Cm can be separated with chelating extractants, e.g., thenoyl-tri-fluor acetone (TTA), benzoyl pyrazolone (PMBP), ethyl-hexyl phosphoric acid (HDEHP) from dilute acids • Am, Cm can be separated with carbamoylmethyl phosphine oxide (CMPO) and TBP containing extractants or ECh resins (TRU resin) or with malonamides or with diglycolamide containing ECh resin (DGA) as ion association complexes. Lanthanides are co-extracted. •Separation of Am, Cm from complex matrices are usually performed after removal other actinides, where Pu, Th and/or U are removed by anion exchangers (AEC) form concentrated HNO_3 or HCl solutions, while Am, Cm get in the effluent. (For Pu removal see the various options above!) From the effluent Am, Cm are pre-concentrated and separated using ECh on TRU resin, or DGA resin. Fe(III) causes significant interference in the retention of Am on TRU resin. • Actinides are pre-concentrated with co-precipitation, e.g. with ferrous hydroxide or Ca fluoride, then actinides after adjustment of the valence states are separated from each other by ECh using TRU resin. •Actinides without pre-concentration, after adjustment of the valence states are separated from each other by ECh using DGA resin. • Am, Cm separation from lanthanides (Ln's) can be performed by anion exchange chromatography. SCN- complexes of Am, Cm and Ln's are separated with dilute HNO_3 and HCl solutions in methanol. • Am, Cm separation from lanthanides (Ln's) can be performed by ECh using TEVA resin. SCN- complexes are separated with elution using formic acid and dilute HCl solutions. • Am, Cm separation from lanthanides (Ln's) can be performed using chelating resin such as HDEHP and complexing agent such as α-HIBA. • Separation of Am from Cm is usually not necessary, but it can be performed by separation after selective oxidation of Am.

	• Am/Cm α source can be prepared by electroplating or micro-precipitation. The previous one is favoured for better resolution.
Chemical yield evaluation	Samples are spiked with Am-243 standard of known activity to determine the chemical recovery of Am in α spectrometry and ICP-MS. Isotope dilution technique is used to calculate the activity of Am-241. It is often assumed that the chemical recovery of Cm is the same as that of Am.
MDA/Detection limit	α spectrometry: 0.1 mBq/sample SF-ICP-MS: Am-241: 0.1 mBq/sample
Sources - detailed information	• Horwitz P., Chiarizia R. Dietz M., Diamond H., Nelson, D.; Separation and preconcentration of actinides from acidic media by extraction chromatography. Analytica Chimica Acta, 281, pp. 361-372 (1993) • Horwitz, E. P. , McAlister, D. R., Bond, A. H., and Barrans, R. E. Jr. (2005). Novel extraction chromatographic resins based on tetraalkyldiglycolamides: Characterization and potential applications. Solvent Extr. Ion Exchange 23, 319-344. • Vajda, N., Kim, C. K., Determination of plutonium isotopes by α spectrometry: A review of analytical methodology, J. Radioanal. Nucl. Chem. 2010, 283, 203-223 • S. K. Aggarwal: Mass spectrometric determination of Am-241, Mass Spectrometry Reviews 2018, 37, 43–56. • E. Holm, S. Ballestra, R. Fukai, A method for ion-exchange separation of low levels of americium in environmental materials, Talanta, 26 (1979) 791 • Vajda, N., Kim, C. K., (2010) Determination of Am-241 isotope by α spectrometry: A review of analytical methodology, J. Radioanal. Nucl. Chem., 284, 341-366 • Horwitz, E. P. (1992). Separation and preconcentration of uranium from acidic media by extraction chromatography. Anal. Chim. Acta 266, 25-37.

2.1.10 U isotopes: U-233, U-234, U-235, U-236, U-238

Property	U isotopes: U-233, U-234, U-235, U-236, U-238
Measurement method	Gamma Spectrometry via detection of daughter nuclides in secular equilibrium with parent: U-238 via Th-234 (63 keV), Pa-234m (131 keV), U-238 and U-234 via Pb-214 (352 keV), Bi-214 (609 keV) Alpha Spectrometry: U-233, U-234, U-235, U-236, U-238 ICP-MS: U-233, (U-234) U-235, U-236, U-238 LSC with extractive scintillator: U-234, U-235, U-238
Property info	Uranium is regarded as major actinide according to the terminology in nuclear industry. U-234, U-235, and U-238 are natural radionuclides, the latter two isotopes are the parents of the two natural decay series with half-lives of billion years. U-235 is a fissile isotope, one of the nuclides responsible for energy production in thermal nuclear reactors operated by U based fuel. It is the major component of some atomic bombs. U-233 is fissile isotope produced by thermal neutron capture of Th-232, it is responsible for energy production in the nuclear reactors operated by Th based fuel. The long-lived U nuclides, i.e., U-233, U-234, U-235, U-236, U-238 are all α emitters. U-236 is produced in the burn out process of U-235, its presence indicates that the sample originates from nuclear fuel or an atomic bomb. These isotopes are not only radiologically important, but they are important in provenance revealing the origin of the sample. Their mass or activity ratios are used in safeguards analysis. The most sensitive technique for the determination of U-234 is α spectrometry, but all U nuclides can be measured by α spectrometry. The highest sensitivity for the measurement the other U nuclides is achieved by mass spectrometric techniques, such as ICP-MS. For the measurement of isotope ratios, various MS techniques can be applied. The natural U-234, U-235, and U-238 isotopes can be easily determined by α spectrometry because the α lines (including those of U-232 tracer) are well separated. The major α lines of U-233 (4824 keV) and U-234 (4777 keV) and those of U-235 (4397 keV) and U-236 (4494 keV) are partially overlapping they cannot be completely resolved in standard α spectrometry, for the accurate determination ICP-MS is used. The amount of U-233 is usually very small in samples from the U fuel cycle, therefore U-233 can be used as radioactive tracer in ICP-MS measurements for U yield determination. U-232 nuclide is an excellent tracer in α spectrometry but not detectable in ICP-MS due to its small specific mass as a result of its short half-life (72 y). Gamma spectrometry can be used in two different ways to detect U isotopes: i) The low energy γ and X-rays can be detected with low energy γ detector, ii) The high energy γ radiation emitted by the isotopes themselves or by daughter nuclides can be detected by γ spectrometry using standard HPGe detectors. Secular equilibrium within the decay series is supposed to exist in the latter technique what is not always attained. U-238 can be detected via short-lived progeny nuclides Th-234 (63,3 and 92,5 keV) and Pa-234m (1001 keV). U-235 can be detected via its 186 keV γ line (54%) but it overlaps with the γ line of Ra-226. These methods are orders of magnitude less sensitive than α spectrometry or ICP-MS. Occasionally U isotopes are detected by LSC most likely by PERALS spectrometry. It has to be mentioned that there are other spectroscopic techniques (AAS, OES, XRF etc.) to detect the element

	uranium, but here no information about the isotope composition is obtained.
Technical info, interferences	• U-234, U-235, and U-238 isotopes are determined by α spectrometry using U-232 tracer. • The major α lines of U-233 and U-234 are overlapping in α spectrum. • The major α lines of U-235 and U-236 are overlapping in α spectrometry. • Accurate determination of U-233 and U-234, as well as that of U-235 and U-236 is made by ICP-MS using U-233 tracer.
Sample preparation	• Reduced U(IV) can be pre-concentrated by co-precipitation with Fe hydroxide, Ca oxalate, Ca fluoride, lanthanide fluorides and hydroxides, MnO_2, $BaSO_4$, Ca phosphate or Ca/Mg carbonate. • U can be separated by LLE using various extractants such as TBP, MIBK after selective removal of other actinides • Separation of U by LLE with TOPO in hexane from 4-6M HCl. It is stripped with dilute acid. • Separation of U with an extractive scintillator containing HDEHP from 0.01 M DTPA solution that masks possible interferences. • Separation of U by AEC using an anion exchange resin from 9M HCl solution. (Other actinides are co-extracted.) U is stripped with dilute acid. • Separation of U by ECh using UTEVA resin containing dipentylpentyl phosphonate from 2-8M HNO_3 or 4-9M HCl. (Other actinides are co-extracted.) U is stripped with dilute acid. • Separation of U by ECh using TRU resin containing TBP-CMPO (Other actinides are co-extracted.) U is stripped with ammonium bioxalate. (U separation is performed after removal of Pu isotopes by AEC from 8M HNO_3). • U sources are prepared by electroplating or micro-co-precipitation with Ln fluorides for α spectrometry after radiochemical separation. • Digestion and oxidation of the sample in order to prepare source for direct measurement by ICP-MS.
Chemical yield evaluation	• In α spectrometry U-232 tracer is used for U yield determination. •In ICP-MS U-233 tracer is used for U yield determination.
MDA/Detection limit	α spectrometry: 0.1 mBq/sample SF-ICP-MS: 1 pg in 1 mL γ spectrometry: 1 Bq/sample
Sources - detailed information	• Horwitz, E. P. (1992). Separation and preconcentration of uranium from acidic media by extraction chromatography. Anal. Chim. Acta 266, 25-37. • Vajda, N., Pöllanen, R., Martin, P., Kim, Ch-K.: Alpha Spectrometry, Chapter 5 in Handbook of Radioactivity Analysis, L'Annunziata (ed.), 2018.

2.1.11 Np-237

Property	Np-237
Measurement method	Alpha Spectrometry ICP-MS NAA via Np-238
Property info	Neptunium is a minor actinide in the terminology of nuclear industry. The most important long-lived (2.14*106 y) isotope of Np is Np-237, it is the only relevant Np isotope of significance in radioactive waste. It is produced by consecutive n reactions, i.e. (n,γ), (n,2n) and radioactive decay from U-235 and U-238 in nuclear reactors and explosions. (The primordial Np-237 has already decayed.) Although the activity of Np-237 is much less than that of Pu-239, and its mass is much smaller than that of U-238, it is of big importance in radioactive waste due to its relatively high mobility. Np-237 is an α emitter with main α particle energy at 4788 keV (51%). It is a DTM nuclide. Np-237 can be measured by α spectrometry, but the main α line overlaps with that of U-234 (4777 keV). Therefore, it can be measured by α spectrometry after separation of the sample matrix and the "complete" removal of U. Much higher sensitivity can be achieved by MS techniques, especially by SF-ICP-MS. The major interference in ICP-MS measurements is the abundance sensitivity due to U-238, therefore high decontamination factors against U have to be assured. AMS is also used for Np measurement because of its ultra-high sensitivity. Np-237 can be detected by NAA via Np-238. The short-lived (T1/2=2.4 d) γ emitter Np-239 is a good tracer to be followed by γ spectrometry for chemical recovery determination. It can be produced by n irradiation of U-238 or by milking from Am-243 tracer. The long-lived (T1/2=150000 y) electron capture Np-236 is a good tracer in ICP-MS measurements.
Technical info, interferences	• The major interference in α spectrometry is U-234 • The major interference in ICP-MS is U-238
Sample preparation	• Np(IV) can be pre-concentrated together with other tetravalent actinides (see Pu isotopes) • Np(IV)is extracted with chelate extractants: TTA in toluene from 1M HNO_3 or HCl, HDEHP from 0.1M HNO_3 • Np(IV) is extracted with ion association complexes: TOPO from 5M HCl • Np(IV) is retained on anion exchange resin or extracted by TOA extractant from 8M HNO_3 or 9M HCl. It is removed with dilute acid (1-4 M HCl) after stripping Th and Pu selectively with 9-10 M HCl, 9-10 M HCl/0.1M NH_4I. • Np(IV) is separated with ECh using TEVA resin, the oxidation state is adjusted using ferrous sulfamate and $NaNO_2$, sample is loaded from 2.5 M HNO_3/0.5 M $Al(NO_3)_3$, Np is stripped with 0.02 M HNO_3/0.02 M HF • Np(IV) can be separated with ECh using UTEVA resin loading from 8M HNO_3 or LN resin from 10 M HCl or TRU resin from 3M HNO_3 or DGA resin from 4M HCl followed by sequential elution of actinides.
Chemical yield evaluation	• Np-239 is a tracer that is detected by γ spectrometry, it is produced by NAA of U-238 or by milking Am-243 • Np-236 is a good tracer for ICP-MS

MDA/Detection limit	α spectrometry: 0.1 mBq/sample ICP-MS: 1 µBq/sample NAA via Np-238: < 0.1 mBq/sample
Sources - detailed information	• Horwitz, E. P., Dietz, M., Chiarizia, R., Diamond, H., Maxwell, S. L., Nelson, M. R. (1995). Separation and pre-concentration of actinides by extraction chromatography using a supported liquid anion exchanger: application to the characterization of high-level nuclear waste solutions. Anal. Chim. Acta 310, 63-78. • Horwitz P., Chiarizia R. Dietz M., Diamond H., Nelson, D.; Separation and preconcentration of actinides from acidic media by extraction chromatography. Analytica Chimica Acta, 281, pp. 361-372 (1993) • Vajda, N., Pöllanen, R., Martin, P., Kim, Ch-K.: Alpha Spectrometry, Chapter 5 in Handbook of Radioactivity Analysis, L'Annunziata (ed.), 2018. • N. Vajda, Zs. Molnár, E. Kabai, Sz. Osvath: Simultaneous Determination of Long-Lived Radionuclides in Environmental Samples, Environmental Radiochemical Analysis II; The Royal Society of Chemistry, Cambridge, 2003; pp. 185-196.) • TRISKEM-International Co: LN resin, https://www.triskem-international.com/scripts/ files/5f46343a7c97d9.45835022/PS_LN-Resin_EN_200603.pdf

2.1.12 Cl-36

Property	Cl-36
Measurement method	LSC AMS
Property info	It is a long-lived ($T_{1/2}$=3.01*10^5 y) radionuclide decaying primarily (98%) by emission of β- electrons (β_{Max} 709 keV) and additionally by electron capture and β+ emission. It is produced from Cl-35 in the Cl-35(n,γ) Cl-36 reaction (target abundance 76 %, cross section σ=10 mb), it is also produced from K-39 by (n,α) reaction (σ=2 b) and from S-34. Cl-36 is present in concrete biological shield, in graphite, it is an impurity in steels and alumina construction materials. Due to its solubility and mobility, it is radiologically important radionuclide as a contaminant in radioactive waste and the environment of nuclear facilities. It is a DTM nuclide that can be measured after chemical separation by LSC at lower (10 mBq/sample) or by AMS at higher sensitivity (1E-15 Cl-36/Cl ratio).
Technical info, interferences	For LSC measurements the sample matrix and most likely all β-emitting radionuclides have to be removed. The removal of other volatile radionuclides such as I-129 is especially important. For AMS measurements stable Cl (Cl-35) is the major interference but for the stability of the chemical processes a Cl carrier is added to the sample. The measuring source in AMS is usually AgCl.
Sample preparation	• Chlorine is separated from the samples as Cl_2 by vaporization that can be performed by heating (pyrolysis above 900°C) or by oxidizing the sample with strong oxidizing agents. • The evaporated Cl_2 can be trapped by graphite cooled with liquid nitrogen or by absorbing the vapours in NaOH solution containing a reducing agent, e.g., $NaNO_2$ that turns chlorine to chloride ions Cl-. • Chloride like iodide ions form insoluble AgCl and AgI precipitate. • Chlorine and iodine have to be separated before LSC measurements. • The separation can be performed by repeated precipitations, selective oxidation and using specific resin: • AgCl precipitate can be selectively dissolved by the addition of ammonia while AgI remains un-dissolved. • Iodide can be oxidized selectively to I2 with H_2O_2 or $NaNO_2$, followed by the extraction of I_2 with CCl_4. • Chlorine can be separated from iodine using the CL resin that is a Pd and Ag selective resin retaining these noble metals partially irreversible. The sample is loaded from 1M H_2SO_4 or 6 mM Na_2CO_3 on the Ag treated CL resin. Chloride is eluted with 0.1M NH_4SCN and iodide is stripped with 0.35M Na_2S. • Chloride can be purified on anion exchange resin. AgCl is dissolved with ammonia and loaded on the column. Chloride ions are eluted with 0.2M NH_4NO_3 – 0.6M NH_4OH.
Chemical yield evaluation	• Chemical recovery of Cl can be determined by measurement of stable Cl by ICP-MS or ion chromatography before and after separation of the sample.
MDA/Detection limit	LSC: 10 mBq/sample AMS: 10-15 Cl-36/Cl ratio

Sources - detailed information	• Hou, X. L., Ostergaard, L. F., Nielsen, S. P. (2007). Analytical procedure for the determination of I-129 and Cl-36 in nuclear waste samples. Anal. Chem., 79, 3126. • TRISKEM-International Co.: CL resin, https://www.triskem-international.com/catalog/ products/resins-and-accessories/cl-resin/bl,product,421,0 • A. Zulauf, S. Happel, M. B. Mokili, A. Bombard, H. Jungclas (2010): Characterization of an extraction chromatographic resin for the separation and determination of Cl-36 and I-129. J. Radanal Nucl Chem, 286(2), 539-546 (DOI: 10.1007/s10967-010- 0772-5) • Inés Llopart-Babot, Mirela Vasile, Andrew Dobney, Sven Boden, Michel Bruggeman, Martine Leermakers, Jixin Qiao, Phil Warwick, 2022. On the determination of 36Cl and 129I in solid materials from nuclear decommissioning activities. Journal of Radioanalytical and Nuclear Chemistry, https://doi.org/10.1007/s10967-022-08327-9.

2.1.13 H-3

Property	H-3
Measurement method	LSC
Property info	This radionuclide can be produced in a reactor by several mechanisms. Neutron capture in deuterium in D_2O moderators is a major source of production in reactors using D_2O. The concrete bioshield is also a source of production from the Li-6(n, α)H-3 reaction with a 953 b cross-section. Tritium decays (half-life: 12.33 a) by β- emission (maximum energy: 19.0 keV), and is a pure β emitter. Tritium in the form of water vapour is extremely mobile in nature and readily exchanges with water in human tissue. The low β energy and the lack of γ emission adds to the difficulty in measuring and assessing levels of tritium in air and other materials. For both H-3 and C-14, the liquid scintillation spectrometer is a well adopted method, and the sample can be mixed directly into a scintillation mixture and counted. In D_2O moderated power and research reactors such as the CANDU type, large quantities of tritium are produced and end up in the moderator systems, heat transport systems, fuel bays, resin tanks, fuel handling systems, etc. Used D_2O will also present a liquid waste disposal problem.
Technical info, interferences	Determination of the H-3 activity concentration of the evaporation residue requires the production of water containing no radioisotopes other than tritium by low temperature vacuum distillation. The purity of the distillate is checked by low background gamma-spectroscopy.
Sample preparation	Low temperature vacuum distillation. The purity of the distillate is checked by low background gamma-spectroscopy. The H-3 activity concentration of the distilled water extracted during digestion is measured using a liquid scintillation (LSC) counter.
Chemical yield evaluation	-
MDA/Detection limit	As low as 0.4 Bq/L
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994). • Review of the different techniques to analyse tritium, Mrs. Pascal FICHET, Arnaud Bultel, Sabina Markelj, Carlos Moreno, TRANSAT - D2.1 - Issued on 2018-07-05 08:53:51 by CEA

2.1.14 C-14

Property	C-14
Measurement method	AMS GPC LSC
Property info	This radionuclide is mainly produced by the activation of trace nitrogen by the N-14(n,p)C-14 reaction with a cross-section of 1.81 b. Additional minor routes are via C-13(n,γ)C-14 from 1.1% abundant C-13 with a cross-section of 0.9 mb and C-12 (98.89%, 3.4 mb) indirectly via C-13. C-14 decays (half-life: 5730 a) by β- emission (maximum energy: 156 keV), and is a pure β emitter. Nitrogen is present in air and in most reactor construction materials and, through activation, generates a significant contribution to the overall radioactive inventory, particularly in concretes and graphite. Assessment of the trace nitrogen levels in construction materials is difficult, posing problems for analysis methods. Hence, reliable input data on nitrogen levels for inventory modelling codes are difficult to obtain and create an increasing necessity to rely on characterization of C-14 by sampling and analysis. The fact that C-14 is a low energy pure β emitter adds to the difficulty in assessing the quantities of this radionuclide. In CANDU reactors, the annulus gap between the pressure and calandria tubes in some situations has been filled with nitrogen gas and air containing nitrogen, resulting in a significant production of C-14 in these components. In addition, large quantities of C-14 occur in the nitrogen cooled graphite moderators of RBMK reactors.
Technical info, interferences	• H-3, in some cases, can contaminate carbon-isolated samples (especially in the use of catalytic furnace), which implies the use of the dual label technique (H-3/C-14) in determination of the activity. Therefore, the efficiency is calculated with quench curves established with H-3 and C-14 certified reference solutions. The window setting optimized is 0-7.0 keV and 7.0-35.0 keV.

Sample preparation	From the liquid waste stream (e.g., evaporation concentrate, ion-exchange resin), the evaporation residue (from primary circuit desorption, decontamination solutions, laboratory waste water) sample is destroyed with concentrated acid (for inorganic component phosphoric acid or sulphuric acid, for inorganic then chromic sulphuric acid), the released carbon dioxide gas is passed through a purification trap containing sulphuric acid (water binding) and bound in an alkaline liquid ($Ba(OH)_2$) in the form of a precipitate ($BaCO_3$). The precipitate ($BaCO_3$) is dried, then reconstituted with hydrochloric acid and the gas formed is absorbed in 3M NaOH, where it is dissolved. The NaOH is then added directly to the HIONIC-FLUOR scintillation cocktail. The C-14 activity concentration of the alkaline trap solution formed during digestion is measured using a liquid scintillation (LSC) counter. Background and standard samples are prepared to have the same quench parameters as the samples to be measured. The carbon content of gas samples is converted to CO_2 gas by catalytic reaction (combustion) with a 450 °C Pt/Pd catalyst. Direct measurement of the released CO_2 gases is possible with a gas proportional counter or, after conversion to graphite, with AMS.
Chemical yield evaluation	GPC: measurement of the amount of produced CO_2 gas AMS: weighing the formed graphite
MDA/Detection limit	AMS: C-14/C-12 ~1.5E-12 GPC: C-14/C-12 ~2.9E-12 LSC: ~40 Bq/Kg C
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994). • NAKAMURA, T. (2004). High precision C-14 measurements with the HVEE Tandetron AMS system at Nagoya University. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms. • Hertelendi, E., Csongor, É., Zaborszky, L., Molnar, J., Gal, J., Györffi, M., & Nagy, S. (1989). A Counter System for High-Precision C-14 Dating. Radiocarbon, 31(03), 399–406. • Arun, B., Vijayalakshmi, I., Ramani, Y., Viswanathan, S., Jose, M. T., Baskaran, R., & Venkatraman, B. (2019). Optimization of C-14 LSC measurement using CO2 absorption technique. Radiochimica Acta, 0(0).

2.1.15 Ca-41

Property	Ca-41
Measurement method	LSC AMS ICP-MS/MS
Property info	It is a long-lived (T1/2=1.03*105 y) radionuclide decaying by electron capture, it emits X-rays (3.3 keV, 11.4%) and Auger electrons (3.0 keV, 77%). It is produced from Ca-40 in the Ca-40(n,γ) Ca-41 reaction (target abundance 97 %, cross section σ=0.4 b). Calcium is major component of concrete, biological shield, soils and sediments, it is an impurity in graphite. Ca-41 is radiologically important radionuclide in decommissioning waste and a possible contaminant in radioactive waste in nuclear facilities. It is a DTM nuclide that can be measured after chemical separation by LSC or AMS.
Technical info, interferences	• The major difficulty in selective separation of Ca is that there are no highly selective separation procedures for Ca. • The major difficulty in LSC measurement is the extremely low energy of X-rays and Auger electrons that fall in the range of electronic noises. A good separation from the matrix and the radionuclides emitting low energy electrons or X-rays is necessary. Another problem arises from the usually high Ca concentration of many sample types.
Sample preparation	The classical procedures are based on many repeated precipitations to obtain a pure Ca fraction. • Ca forms carbonate precipitate together with Sr and Ba, while alkali metals remain dissolved and can be separated. • Ca is precipitated as $Ca(OH)_2$ together with the hydroxides of Sr, Ba from NaOH solutions of 0.5M or higher. • Ca is separated more selectively as $Ca(OH)_2$ from NaOH solutions of pH 9 while Sr, Ba remain in the filtrate. These two separation steps are combined and partially separated. • Ba can be separated from Ca as Ba chromate precipitate. • Sr and partially Ba can be separated by retaining them on Sr resin from ≥ 3M HNO_3 while Ca gets in the effluent. • Additionally $Fe(OH)_3$ precipitations are performed to remove various contaminants such as actinides, lanthanides, Fe, Co, Ni etc. by scavenging. Many contaminants can be removed by anion exchange chromatography loading the sample from 8M HNO_3 and/or 9M HCl. The use of TRU resin for further purification of Ca from actinides and lanthanides was also proposed.
Chemical yield evaluation	• For measurement of the chemical recovery a Ca-45 tracer can be used that is a pure β emitter (β_{Max} 257 keV) and its half-life is 163 days. It can be measured by LSC after correction for spectrum overlapping. Chemical recovery of Ca can be determined by measurement of stable Ca in the sample by AAS, ICP-OES, ICP-MS before separation and afterwards.
MDA/Detection limit	LSC: ~10-1 Bq/g Ca using 1 g soil/concrete AMS: 10-6- 10-8Bq/g Ca ICP-MS/MS: 0.32 Bq g^{-1} (0.099 ng g^{-1})

Sources - detailed information	• Hou, X. (2005): Separation procedure for the determination of Ca-41 in concrete. Radiochim. Acta, 93, 611. • Hampe, D., Gleisberg, B., Akhmadaliev, S. et al. (2013). Determination of Ca-41 with LSC and AMS: method development, modifications and applications. J Radioanal Nucl Chem 296, 617–624. • Russell, B., Mohamud, H., Miranda, M.G., Ivanov, P., Thompkins, H., Scott, J., Keen, P., Goddard, S., 2021. Investigating the potential of tandem inductively coupled plasma mass spectrometry (ICP-MS/MS) for 41Ca determination in concrete. J. Anal. At. Spectrom. 36, 845–855. https://doi.org/10.1039/d1ja00027f

2.1.16 Mo-93

Property	Mo-93
Measurement method	LSC ICP-MS AMS
Property info	It is a long-lived (T1/2=4000 y) radionuclide that decays to Nb-93m (88%) and to Nb-93 (12%) by electron capture, it does not emit easy to measure γ radiation. In the decay process Auger electrons and X-rays of 16,5 keV (21%), 16,6 keV (41%), 18,6 keV (9%) are emitted. It is produced from Mo-92 in the Mo-92(n,γ) reaction (target abundance 14.8 %, cross section about σ=0.45 mb). Molybdenum is an alloying metal of steels (0.25-1.7%) one of the main structural materials of nuclear reactors. It is a DTM nuclide that can be measured by X-ray spectrometry, by LSC via Auger electrons and X-rays, and by mass spectrometry. Radiochemical separation has to be performed before measurement to separate Mo from other radionuclides in LSC, from Nb (in particular Nb-93m) in X-ray spectrometry and stable Nb as major isobaric interferences together with radioactive Zr-93 in ICP-MS. Molybdenum can exist in various oxidation states, the most stable in aqueous solutions being +IV and +VI. It can form insoluble MoO_2 and MoO_3. Molybdenum oxides can be dissolved in alkaline solutions as MoO_4^{2-} molybdate anions and in HF and H_2SO_4. It can be reduced to Mo(III), Mo(IV) and Mo(V). Molybdate ions are stable in alkaline solutions while in acidic solutions polymolybdates and molybdic acid are formed.
Technical info, interferences	Since Nb-93m is produced by not only the EC decay of Mo-93 but also the β-decay of Zr-93, Mo has to be separated from Zr and Nb to determine Mo-93 using the X-rays. Moreover, the X-ray energy of Mo-93 is sufficiently low to be absorbed by the sample matrix and/or glass vial; further, the chemical separation of Mo from the matrix elements and the preparation of thin-layer source are indispensable.

Sample preparation	For the selective separation of Mo LLE, anion exchange and absorption on alumina are used often with further purification steps such as precipitations and cation exchange to remove cationic interferences. Molybdenum together with Nb is extracted from 4M HCl/1M $AlCl_3$ with α-benzoinoxime (ABO) while Zr gets in the effluent. In the method of Bombard, Mo and Nb are back-extracted with 4M NH_4OH and further separated by precipitation. Anionic molybdate species together with Zr, Nb anions can be retained on anion exchangers or can be extracted by the quaternary amine containing ECh resin, i.e. TEVA resin anion exchangers or can be extracted by the quaternary amine containing ECh resin, i.e. TEVA resin from HF from HF or HCl solutions. The retention of Mo on alumina (Al_2O_3) in diluted HNO_3 (pH=3.0±0.5) is widely used in Mo-99/Tc-99m-generators. Mo is also retained from more concentrated HNO3, or diluted (≤0.1 M) HF, and it can be eluted (together with other anions) from alumina with alkaline solutions. For the determination of Mo-93 in radioactive waste Osvath et al. combined the ECh procedure on TEVA resin with the purification of Mo on alumina followed by LSC measurement. AMS: Decomposition of 3 – 4 g steel sample in HNO_3/HCl/HBF_4 - Evaporate to dryness Dissolve residue in 2M HF Load onto anion exchange column DOWEX 1 X 8 1. Elute with 2M HF : elute iron 2. Elute with 8M HNO_3 : elute molybdenum Evaporate HNO_3 eluate to dryness and redissolve in conc. NH_3 Evaporate solution and transform the residue to the oxide AMS measurement of the sample
Chemical yield evaluation	ICP-AES
MDA/Detection limit	AMS: Mo-93/Mo-92 of <2E-9 ICP-MS: 0.2 Bq/g

Sources - detailed information	• Horwitz, E. P., Dietz, M., Chiarizia, R., Diamond, H., Maxwell, S. L., Nelson, M. R. (1995). Separation and preconcentration of actinides by extraction chromatography using a supported liquid anion exchanger: application to the characterization of high-level nuclear waste solutions. Anal. Chim. Acta 310, 63-78. • Bombard, A.: Determination of the long-lived radionuclides Zr-93, Mo-93 and Nb-94 in nuclear waste. PhD thesis, Universite de Nantes, 2005. • Development of HCl-free solid-phase extraction combined with ICP-MS/MS for rapid assessment of difficult-to-measure radionuclides. Part I. Selective measurement of Zr-93 and Mo-93 in concrete rubble Van-Khoai Do; Takahiro Furuse; Erina Murakami; Rena Aita; Yuki Ohta; Soichi Sato • Shimada A, Kameo Y (2016) Development of an extraction chromatography method for the analysis of Zr-93, Nb-94, and Mo-93 in radioactive contaminated water generated at the Fukushima Daiichi Nuclear Power Station. J Radioanal Nucl Chem 310:1317-1323 • Osváth, Sz.: Determination of Mo-93 (and Nb-94) in nuclear decommissioning waste from a nuclear reactor, NKS-B RadWorkshop, DTU Riso, Oct 8-12, 2018 • Measurement of the Activation Product Mo-93 via AMS G. Rugel, T. Faestermann, A. Inserra a, G. J¨org a, G. Korschinek, and C. Lierse von Gostomski a a Institut fur Radiochemie, TU M¨unchen, Walther-Meissner-Strasse 3, 85748 Garching • Shimada, A., Ohmori, H., & Kameo, Y. (2017). Development of determination method of Mo-93 content in metal waste generated at the Japan Power Demonstration Reactor. Journal of Radioanalytical and Nuclear Chemistry, 314(2), 1361–1365.

2.1.17 Tc-99

Property	Tc-99
Measurement method	LSC ICP-MS
Property info	It is an almost pure β- emitting isotope of maximum β energy in the medium energy range (βMax 294 keV). It is produced by high fission yield (6%), and is also an activation product of Mo-98, therefore it has relatively high activity concentration in radioactive waste. Due to its very long half-life (T1/2=2.11*105 y) and high mobility it represents a long term, high radiation risk. It is a DTM nuclide, for its determination by LSC, other β counting techniques, or by means of ICP-MS radiochemical separation is required to remove the matrix components and interfering radionuclides/elements including the chemically similar Ru isotopes (Ru-106 with half-life of 374 d) and stable Ru containing Ru-99 (abundance 12.7%) and Ru-100 (abundance 12.6%). The latter isotopes are responsible for isobar interference and abundance sensitivity, respectively in ICP-MS. Typical detection limits in β and LSC measurement are 1-100 mBq/sample. ICP-MS is more sensitive allowing measurements at detection limit of 1 µBq/sample. Tc has no stable isotope, therefore, it has no isotopic carrier, but Re is often used as non-isotopic carrier. In solutions Tc forms pertechnetate TcO4- anions, that are responsible for the high mobility of Tc in nature, however it can be reduced readily to Tc(IV), e.g. at pH 4 with potassium pyrosulfite ($K_2S_2O_5$) and Fe2+.
Technical info, interferences	Radiochemical separation is required to remove the matrix components and interfering radionuclides/elements including the chemically similar Ru isotopes (Ru-106 with half-life of 374 d) and stable Ru containing Ru-99 (abundance 12.7%) and Ru-100 (abundance 12.6%). The latter isotopes are responsible for isobar interference and abundance sensitivity, respectively in ICP-MS. Tritium is co-extracted with fraction of Tc-99; however, this interference can be avoided using dual technique in the determination by LSC.

Sample preparation	Tc can be pre-concentrated by co-precipitation with $Fe(OH)_2$ as TcO_2 when pH is 9 or higher. TcO_2 can be dissolved with HCl and oxidized back to TcO_4^- with H_2O_2. Tc can also be concentrated on an anion exchange resin from low acidity solutions. $RuCl_4^-$ also retained on the resin can be selectively removed by oxidation with NaOCl. Tc can be separated by LLE, anion exchange and ECh. The principle of most procedures is the formation of strong complexes of TcO_4^- with quaternary amines, e.g., in the form TOA that is extracted with xylene or strong basic anion exchange resins, or TEVA resin. TcO_4^- anions are strongly bound from dilute acid and can be stripped/eluted with more concentrated acids or NaOH. With this procedure a good separation from tetravalent actinides is achieved. Examples for the separation are the following: TcO_4^- is extracted with TOA/xylene from sulfuric acid and back extracted with 2M NaOH. TcO_4^- is retained from 0.1M nitric acid (containing H_2O_2) on TEVA resin, it is stripped with 12M HNO_3. Recently a TOA impregnated membrane, the TK201 disc has been prepared for the rapid concentration and separation of Tc from water samples where sample is loaded from 0.01M HNO_3 containing H_2O_2 and Tc is stripped with 2M NH_4OH. The new ECh material containing polyethyleneglycol coated resin, the TK202 resin can retain Tc from 5M NaOH solution and Tc is stripped with distilled water. The complete separation methods usually contain pre-concentration, separation, and purification steps together with source preparation.
Chemical yield evaluation	To determine the chemical recovery the short-lived (T1/2= 6 h) Tc-99m tracer emitting 140 keV (89%) γ radiation can be used. If the sample is measured by LSC a longer cooling time is needed before measurement for the decay of the tracer. The non-isotopic Re carrier is also often measured by ICP-MS.
MDA/Detection limit	Typical detection limits in β and LSC measurement are 1-100 mBq/sample. ICP-MS is more sensitive allowing measurements at detection limit of 1 µBq/sample.
Sources - detailed information	• Horwitz, E. P., Dietz, M., Chiarizia, R., Diamond, H., Maxwell, S. L., Nelson, M. R. (1995). Separation and preconcentration of actinides by extraction chromatography using a supported liquid anion exchanger: application to the characterization of high-level nuclear waste solutions. Anal. Chim. Acta 310, 63-78. • TRISKEM-International Co: TK201 disc, https://www.triskem-international.com/scripts/ files/624a3ab0aba5d6.17415344/PS_TK201-Resin_EN_220330.pdf • TRISKEM-International Co: TK202 resin, https://www.triskem-international.com/scripts/ files/616ebadb007443.55241683/PS_TK202-Resin_FR_210816.pdf • Shi, K., Hou, X., Roos, P., & Wu, W. (2012). Determination of technetium-99 in environmental samples: A review. Analytica Chimica Acta, 709, 1-20. https://doi.org/ 10.1016/j.aca.2011.10.020

2.1.18 I-129

Property	I-129
Measurement method	LSC NAA ICP-MS AMS Gamma spectrometry
Property info	It is a very long-lived ($T_{1/2}$=1.57*10^7 years) β- decaying isotope of maximum β energy in the low energy range (β_{Max} 154 keV). Its daughter nuclide Xe-129m decays by internal conversion to Xe-129 and emits low energy, low intensity γ radiation (40 keV, 7.5%) and conversion and Auger electrons. I-129 is a fission product of fission yield of 0.84%. Due to its long half-life and high mobility, it is a radiologically very important radionuclide in waste samples in the long term. It is a DTM nuclide that can be measured by low energy γ spectrometry, LSC, NAA, ICP-MS, and AMS. For sensitive measurements radiochemical separations are required for each measurement method. Detection limits vary in the wide range of 100 mBq/sample (γ spectrometry) and nBq/sample (AMS). High sensitivities of about 1 µBq/sample are achieved with RNAA where I-130 is produced by neutron absorption and detected via its γ radiation (536 keV, 99%; 668 keV, 96%). ICP-MS is less sensitive. The major interferences in the radiometric methods are other radioactive nuclides, in ICP-MS and AMS stable iodine (I-127). The sample matrix has to be removed in all DA techniques. The same radiochemical separation procedures can be applied for all measurement techniques, but the final measurement sources are different. In γ spectrometry, it can be a PdI_2 precipitate, in LSC and ICP-MS the source is iodide in liquid that is mixed with a scintillation cocktail for LSC, in RNAA the source can be MgI_2 or PbI_2, and in AMS the source is AgI.
Technical info, interferences	The major interferences in the radiometric methods are other radioactive nuclides, in ICP-MS and AMS stable iodine (I-127). The sample matrix has to be removed in all DA techniques.
Sample preparation	Iodine can be separated from solid samples as I_2 using oxidizing agents such as H_2SO_4/HNO_3 or by combustion. The evaporated I_2 can be trapped in NaOH solution containing a reducing agent, e.g., $NaNO_2$ that turns iodine to iodide ions I-. Iodide can be obtained from solid samples by leaching. Iodide ions form insoluble AgI precipitate. The separation is most frequently performed by selective oxidation and reduction cycles in combination with extraction. Iodide can be oxidized selectively to I_2 with H_2O_2 or $NaNO_2$, followed by the extraction of I_2 with CCl_4. Iodide can be back-extracted in aqueous phase with a reducing agent such as $NaHSO_3$. This procedure will separate Cl^- ions, as well. Iodide and chloride can be separated using the CL resin that is a Pd and Ag selective resin retaining these noble metals partially irreversibly. The sample is loaded from 1M H_2SO_4 or 6 mM $NaCO_3$ on the Ag treated CL resin. Chloride is eluted with 0.1M NH_4SCN and iodide is stripped with 0.35M Na_2S. Iodide can be purified on anion exchange resin. Sample is loaded from 0.01M HNO_3 on the column. Iodide ions are eluted with 2M $NaNO_3$.

NAA:
Solid sample - Alkali fusion/ashing, water leaching or combustion and trapping iodine with KOH solution
Leachate or trap solution or water sample as starting point
Convert all iodine species to iodide, extraction of iodine with CCl_4 and back extraction with H_2SO_3
Add MgO_2 to back extracted solution, dry to convert iodine to MgI_2, sealed in quartz ampoule
Neutron irradiation in reactor
Break the ampoule, dissolve the irradiated sample with HCl with $NaHSO_3$
Separation of iodine by CCl_4 extraction, back extracted iodide is precipitated as PdI_2, filtered
Gamma spectrometry for measurement of I-130 and I-126

ICP-MS:
Same as NAA

TIMS:
The target materials for isotopic measurements are loaded in the instruments in solid-phase without solvent and are subsequently atomized, evaporated, and ionized on the hot surface of the filaments.

AMS:
Any type of sample is applicable for I-129 isotopic measurements using AMS as long as the final chemical form can be prepared as AgI, which can be pressed on a tantalum or copper target.
AMS covers the iodine contained samples of particulate matter, aerosols, organisms, nuclear materials, soil, sediments, and water.
For quantification of I-129, a carrier (mostly as sodium iodide or potassium iodide) with the natural abundance isotopic ratio of iodine is added to the samples before iodine dissolution and extraction.
Gaseous I-129 and the aerosols containing I-129 are collected by triethylene diamine (TEDA) activated charcoal filters and glass microfiber filters, respectively.
The filters are mixed with sodium hydroxide (NaOH) and sodium hydrogen sulfite ($NaHSO_3$) solution to dissolve iodine as iodide (I-) followed by purification of iodine using selective extraction by carbon tetrachloride (CCl_4), dichloromethane (CH_2Cl_2), chloroform ($CHCl_3$), or hexane (C_6H_{14}). The purified iodine is then back extracted into aqueous $NaHSO_3$ solution. The charge state of iodine is controlled to avoid iodine loss as I_2. Further, SO_3^{-}, and SO_4^{2-} in the solution are removed by adding barium nitrate ($Ba(NO_3)_2$) then separating the corresponding precipitates, ($Ba(SO_3)_2$ and $BaSO_4$). The purification procedure can be modified using an ion–exchange column to avoid the use of toxic CCl_4 or $CHCl_3$. Finally, iodide is precipitated to form AgI by adding sodium nitrate ($AgNO_3$) to the solution. The iodine collected in the charcoal filters can also be extracted as gaseous I_2 by adding nitric acid (HNO_3) to the filters in an Erlenmeyer flask placed in a metal bead bath heated to 85 °C. The extracted I_2 is then introduced to a bottle containing $AgNO_3$ solution, in which I_2 reacts with Ag^+ to form AgI

	precipitate. A different iodine dissolution procedure is required to prepare biological (algae, lichens, corals), environmental (soils, sediments, and rocks), spent resin (radioactive resin waste), and nuclear waste samples to measure their I-129 content. The iodine content is digested in HNO_3 solution, usually by leaching or combustion methods. However, the digestion time and probability of cross-contamination during the sample preparation are reduced by applying the microwave digestion method. Iodine purification using $CHCl_3$ and $NaHSO_3$, and precipitation using $AgNO_3$ follow the above-mentioned procedure. However, the iodine dissolution procedures are not required for analyzing I-129 concentration of water (including seawater) samples. After filtering microparticles and/or converting organic iodine to inorganic iodine by adding potassium persulfate ($K_2S_2O_8$), either an ion-exchange method or solvent extraction, back-extraction method using $CHCl_3$ or CH_2Cl_2, and $NaHSO_3$ are applied for separation and purification of iodine, followed by AgI precipitation.
Chemical yield evaluation	The chemical recovery can be determined by measuring I-125 tracer that is an electron capture nuclide with half-life of 59 days. It emits low energy γ radiation (35.5 keV, 6.7%) and several X-rays. Stable iodine (I-127) as a carrier can also be measured by ICP-MS. From stable iodine I-126 is produced in (n,2n) reaction that can be measured together with I-130 in RNAA
MDA/Detection limit	LSC: Detection limit: 10 mBq NAA: Detection limit of 1 microBq (or 2 × 10−13 g, or 109 atoms, or I-129/I-127 ratio of 10−10) ICP-MS: Isotope ratio: 1E-5~1E-8; Concentration: < ~ 80 ng/L AMS: The detection limits for the concentration and isotopic ratio are as low as 105 atoms/g and 1E-15, respectively. TIMS: The detection limits for the concentration and isotopic ratio are as low as 6 ppb and 1E-8, respectively. Gamma spectrometry: Detection limit: 20–200 mBq
Sources - detailed information	• TRISKEM-International Co.: CL resin, https://www.triskem-international.com/catalog/ products/resins-and-accessories/cl-resin/bl,product,421,0 • Lehto, J., Hou, X. (2010) Radiochemistry of the halogens, in Chemistry and Analysis of Radionuclides, pp. 181-191, Wiley-VCH, Germany • Kim, J., Kim, J-Y., Bae, S-E., Song, K., Park, J-H. (2021) Review of the development in determination of I-129 amount and the isotope ratio of I-129/I-127 using mass spectrometric measurements. Microchem. J. 169,106476. • X. Hou, V. Hansen, A. Aldahan, G. Possnert, O.C. Lind, G. Lujaniene, A review on speciation of iodine-129 in the environmental and biological samples, Anal. Chim. Acta 632 (2009) 181-196.

2.1.19 Mn-54

Property	Mn-54
Measurement method	Gamma spectrometry
Property info	This radionuclide is produced primarily by the Fe-54(n,p)Mn-54 reaction, which has an average cross-section, in a fission neutron spectrum, of 53 mb and decays (half-life: 312 d) via electron capture and 835 keV γ emission. The target isotope, 5.8% abundant Fe-54, is present in the steel construction materials of the pressure vessel, fuel support structures and the primary circuit. Corrosion of the steel components can transport iron to the fast neutron flux region of the reactor where Mn-54 is produced. Because of its relatively short half-life, Mn-54 can be significant only briefly after reactor shut- down. It can be measured by γ spectrometry.
Technical info, interferences	-
Sample preparation	Can be directly measured by gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	6.4 [1.6–15.7] mB/g
Sources - detailed information	• IAEA-TRS No. 389: Radiochemical Characterization of Shut Down Nuclear Reactors for Decommissioning Purposes, 1998 • Radiological characterisation for the clearance of burnable waste produced at CERN Richard Harbron, Renaud Charousset , Gerald Dumont , Matteo Magistris , Nabil Menaa , Paolo Giunio Pisano , Christian Theis, Applied Radiation and Isotopes 196 (2023) 110782

2.1.20 Fe-59

Property	Fe-59
Measurement method	Gamma spectrometry
Property info	Radioactive Fe-59 has been produced through the reactions Fe-58(d, p)Fe-59 and Co-59(n, p)Fe-59 the half-life is 47+/-3 days. The emitted particles are negative electrons, most of which have a range of 0.09 grams/cm^2 Al, while a small number extend to 0.35 grams/cm^2 Al.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	~1.3 Bq/kg
Sources - detailed information	• Jiaying Wang, Yu Chen, Zhongjian Ma, Lu Zhang, Zhen Zhang, Analysis of gamma-emitting radionuclides in soils around high energy accelerators, Radiation Medicine and Protection, Volume 3, Issue 4, 2022, Pages 171-174, ISSN 2666-5557

2.1.21 Co-58

Property	Co-58
Measurement method	Gamma spectrometry
Property info	Radioisotope Co-58 is one of the radioisotopes which is developed for the preparation of labelled compounds, it has a short half- life of T1/2 = 71 days (gamma energy: 811 keV). Radioisotope Co-58 has a gamma radiation energy which is suitable for the radiotracer technique purpose. Radioisotope Co-58 can be produced through neutron activation reaction Ni-58(n,p)Co-58 in a nuclear reactor by irradiation the NiO target.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	6.1 [1.5–14.4] mBq/g
Sources - detailed information	• Radiological characterisation for the clearance of burnable waste produced at CERN Richard Harbron, Renaud Charousset , Gerald Dumont , Matteo Magistris , Nabil Menaa , Paolo Giunio Pisano , Christian Theis, Applied Radiation and Isotopes 196 (2023) 110782

2.1.22 Co-60

Property	Co-60
Measurement method	Gamma spectrometry
Property info	This radionuclide is produced by the Co-59(n,γ)Co-60 reaction in the 100% abundant stable cobalt isotope Co-59 with a cross-section of 18.7 b. Co-60 decays (half-life: 5.27 y) by β- emission (maximum energy: 318 keV) to excited levels of Ni-60 and produces two major γ rays: 1.17 MeV and 1.33 MeV. Cobalt is a trace constituent in both carbon and stainless steels (ranging from 80 to 150, and 230 to 2600 ppm, respectively). Cobalt is also present in Inconel and Monel. This isotope is the dominant dose producing radionuclide in the reactor interior on a 10 to some 50-year timescale. The production rate of Co-60 is sufficiently high in the high flux region near the core that a substantial portion of the stable cobalt (up to one third) may be transmuted over the life of the reactor. Deposition of Co-60 from the corrosion of ferritic materials is a well-known problem in GCRs. After production in the reactor core, translocation and deposition of Co-60 throughout the reactor systems will be a function of (1) corrosion controls, (2) the effectiveness of the reactor coolant cleanup system, and (3) the radioactive waste management practices. It is easily measured by γ spectrometry.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	10 Bq/l
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994).

2.1.23 Se-79

Property	Se-79
Measurement method	AMS LSC ICP-MS
Property info	Se-79 is a long-lived fission product with chemical and radiological toxicity. It is one of the radionuclides of interest in nuclear waste disposal due to its potential migration capacity to the surface environment.
Technical info, interferences	One of the major obstacles in the measurement of ultratrace Se-79 with AMS is the strong interference from the isobaric nuclide Br-79.
Sample preparation	• The procedure for sample preparation involves four steps: SeO_2 (with or without Se-79 spike) is dissolved in 10 ml deionized water, and the PH value is adjusted to 6.8 with 2 M NaOH • The Ag_2SeO_3 was precipitated by adding slightly excessive $AgNO_3$ solution • The precipitation is then centrifugalized after cooling at 4 °C for 4 h in a refrigerator. • The Ag_2SeO_3 precipitation is washed with cold deionized water and centrifuged for three cycles to remove the residual SeO_2 and ensure the quality of the extracted ion beam. • The Ag_2SeO_3 powder is dried at 100 °C for 12 h in an oven, and then baked at 320 °C for 1 h in a muffle furnace for further removal of SeO_2 . • The produced white Ag_2SeO_3 is stored in a desiccator.
Chemical yield evaluation	Gravimetrically by weighing using stable Se
MDA/Detection limit	Sensitivity for Se-79/Se ratio is better than 1E -12, equivalent to a detection limit of femtomole (1E-15 mol) Se-79 in a milligram-sized sample.
Sources - detailed information	• Wang, W., Guan, Y., He, M., Jiang, S., Wu, S., & Li, C. (2010). A method for measurement of ultratrace Se-79 with accelerator mass spectrometry. Nuclear Instruments and Methods in Physics Research Section B: Beam Interactions with Materials and Atoms, 268(7-8), 759–763. • Aguerre S, Frechou C (2006) Development of a radiochemical separation for selenium with the aim of measuring its isotope 79 in low and intermediate nuclear wastes by ICP-MS. Talanta 69:565–571. https://doi.org/10.1016/j.talanta.2005.10.028 • Jörg G, Bühnemann R, Hollas S, Kivel N, Kossert K, Van Winckel S, Gostomski CL v. (2010) Preparation of radiochemically pure 79Se and highly precise determination of its half-life. Appl Radiat Isot 68:2339–2351. https://doi.org/10.1016/j.apradiso.2010.05.006

2.1.24 Zr-95

Property	Zr-95
Measurement method	Gamma spectrometry
Property info	Zr is commonly used in nuclear reactors for its ability to withstand high temperatures and its low neutron-capture cross-section. Zr-95 decays to Nb-95 by β- emission (maximum energy: 1.12413 MeV) with a half-life of 64 days.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	~1.2 Bq/kg
Sources - detailed information	• Jiaying Wang, Yu Chen, Zhongjian Ma, Lu Zhang, Zhen Zhang, Analysis of gamma-emitting radionuclides in soils around high energy accelerators, Radiation Medicine and Protection, Volume 3, Issue 4, 2022, Pages 171-174, ISSN 2666-5557

2.1.25 Ru-106

Property	Ru-106
Measurement method	Gamma spectrometry
Property info	Ru-106 is produced by fission and decays (half-life: 374 d) by β– emission (maximum energy: 39 keV) to Rh-106. Rh-106 decays (half-life: 30 s) by β– emission (maximum energy: 3.54 MeV) to stable Pd-106. The main γ emitters are from the Rh-106 decay at 512 and 622 keV. Ruthenium can be present in different valencies and can form volatile species in high temperature conditions. In solution, ruthenium can be present as unstable species and deposit on metallic surfaces, forming quite strongly adherent layers. Owing to its short life, Ru-106 is not a radionuclide critical for disposal. Ru-106 can cause some radiation hazards by formation of hot spots, mainly in reprocessing or high-level waste treatment facilities. Ru-106 can easily be measured by γ spectrometry via Rh-106 as Ru-106 itself has no gamma lines.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	Detection limit: 5-10 µBq/m3
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994). • H. Ramebäck, C. Söderström, M. Granström, S. Jonsson, J. Kastlander, T. Nylén, G. Ågren, Measurements of Ru-106 in Sweden during the autumn 2017: Gamma-ray spectrometric measurements of air filters, precipitation and soil samples, and in situ gamma-ray spectrometry measurement, Applied Radiation and Isotopes, Volume 140, 2018, Pages 179-184, ISSN 0969-8043

2.1.26 Ag-108m

Property	Ag-108m
Measurement method	Gamma spectrometry
Property info	This radionuclide is produced by the Ag-107(n,γ)Ag-108m reaction in the 51.8% abundant Ag-107. Ag-108m decays (half-life: 130 a) by electron capture (weak X rays) to Ag-108, which decays (half-life: 2.4 min) by β– emission (maximum energy: 1.655 MeV) to stable Cd-108. The low cross-section (0.33 b) of Ag-107 and the long half-life of Ag-108m limit its production; nevertheless, the use of large amounts of silver in PWR control rods will result in a large inventory of Ag-108m in these components. Ag-108m is measured by γ spectroscopy of multiple energies or by the equilibrium β– decay of Ag-108.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	1 Bq/l
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994).

2.1.27 Ag-110m

Property	Ag-110m
Measurement method	Gamma spectrometry
Property info	This radionuclide is produced by the 109Ag(n,γ)Ag-110m reaction in the 48.2% abundant stable isotope. Ag-110m decays (half-life: 249 d) by β– emission (maximum energy: 1.467 MeV) to Ag-110, which decays (half-life: 24.5 s) by β– emission (maximum energy: 2.893 MeV) to stable Cd-110. The relatively large abundance of Ag-110m can be explained by the presence of silver– indium–cadmium control rods which, like other reactor components, are subject to corrosion and erosion processes that can lead to contamination of the primary coolant. Traces of Ag-110m can also be observed in some reactors in which a silver alloy was used for sealing the head of the reactor pressure vessel. Silver at the ppb level in Magnox GCRs is one controller of the long term γ field for long deferment periods. It is measured by γ spectrometry at multiple energies or by the equilibrium β– decay of Ag-110.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	6.3 [3.5–9.1] mBq/g
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994). • Radiological characterisation for the clearance of burnable waste produced at CERN Richard Harbron, Renaud Charousset , Gerald Dumont , Matteo Magistris , Nabil Menaa , Paolo Giunio Pisano , Christian Theis, Applied Radiation and Isotopes 196 (2023) 110782

2.1.28 Sn-113

Property	Sn-113
Measurement method	Gamma spectrometry
Property info	Sn-113 is produced through (n, γ) reactions or as daughter of Cd-113m in nuclear reactors.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	Information needed
Sources - detailed information	• INTERNATIONAL ATOMIC ENERGY AGENCY, Strategy and Methodology for Radioactive Waste Characterization, IAEA-TECDOC-1537, IAEA, Vienna (2007)

2.1.29 Cs-134

Property	Cs-134
Measurement method	Gamma spectrometry
Property info	This radionuclide is produced by the Cs-133(n,γ)Cs-134 reaction from Cs-133, which is either a fission product or the only stable isotope of natural caesium. From the viewpoint of decommissioning, Cs-134 can therefore appear as a contaminant on various reactor components or as an activation product mainly in concrete structures. Cs-134 decays (half-life: 2.065 years) by β– emission (maximum energy: 658 keV) or by electron capture to stable Ba-134. The decay processes produce also several γ with high energies (e.g., 605 and 796 keV). Cs-134 can easily be measured by γ spectrometry. In case of prompt dismantling, Cs-134 must be taken into account both as an ingestion hazard and as a source of external radiation.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	Information needed
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994).

2.1.30 Cs-137

Property	Cs-137
Measurement method	Gamma spectrometry
Property info	This radionuclide is produced by fission and is one of the most abundant fission products. Cs-137 decays (half-life: 30 y) by β– emission (maximum energy: 1.17 MeV) to Ba-137. Approximately 85% of the β decays are through Ba-137m and thus are accompanied by the emission of its 662 keV photons. Barium X rays and conversion electrons are also emitted. Because of its high water solubility, Cs-137 is easily transported in most LWR circuits. Being a volatile isotope, it may cause inhalation hazards to the decommissioning work force. The design life of disposal facilities (300 a) is based on the Cs-137 half-life. Cs-137 is easily measured by γ spectrometry (Even though Cs-137 primarily undergoes beta decay, the gamma radiation emitted during its decay process can be measured and quantified by gamma spectrometry → via decay of Ba-137m to its ground state).
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	6.2 [3.1–9.4] mBq/g
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994). • Radiological characterisation for the clearance of burnable waste produced at CERN Richard Harbron, Renaud Charousset , Gerald Dumont , Matteo Magistris , Nabil Menaa , Paolo Giunio Pisano , Christian Theis, Applied Radiation and Isotopes 196 (2023) 110782

2.1.31 Ce-144

Property	Ce-144
Measurement method	Gamma spectrometry
Property info	Ce-144 is produced by fission and decays (half-life: 285 d) by β– emission (maximum energy: 318 keV) to Pr-144. Pr-144 decays (half-life: 17 min) to a stable element. Approximately 76% of the emitted b particles have a maximum energy of 318 keV and 20% have a maximum energy of 185 keV. Approximately 11% of the beta decays are accompanied by the emission of a 133 keV gamma photon. Because of its short life, Ce-144 is not a radionuclide critical for disposal. Although it is analysed as a γ emitter, optimum precision and accuracy are reduced by interferences. A wet radiochemistry procedure is available to obtain a pure cerium fraction which can be measured on a γ spectrometer.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	Information needed
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994).

2.1.32 Eu-152

Property	Eu-152
Measurement method	Gamma spectrometry
Property info	Eu-152, Eu-154 and Eu-155 are produced by neutron capture in Eu-151 (47.8%) and Eu-153 (52.2%). Other routes to the production of Eu isotopes occur because of chain absorptions in Sm. Eu-152 decays (half-life: 13.5 a) by β– emission (maximum energy: 1.477 MeV) to Gd-152, which decays (half-life: 1.1×10^{14} y) by α emission. Eu-154 decays (half-life: 8.6 a) by β– emission (maximum energy: 1.85 MeV) to stable Gd-154. Eu-155 decays (half-life: 4.76 a) by β– emission (maximum energy: 2.52 keV) to stable Gd-155. Eu-152 and Eu-154 are the two dominant europium activation products in bioshield concrete and core graphite on a timescale of 10–20 years. Both have very large neutron capture cross-sections; Eu-152 is produced primarily by thermal neutrons, whereas Eu-154 also has a substantial resonance integral. The activities of Eu isotopes require consideration, owing to the presence of trace quantities of rare earth elements in source materials used in reactor graphite and bioshield concretes. Typically, sub-ppm levels of Eu and Sm parents generate sufficient activity on neutron activation to warrant precautionary measures during the early decommissioning of concrete bioshields. These Eu isotopes are measured by γ spectrometry at multiple energies or by β counting.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	15.9 [5.5–33.1] mBq/g
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994). • Radiological characterisation for the clearance of burnable waste produced at CERN Richard Harbron, Renaud Charousset , Gerald Dumont , Matteo Magistris , Nabil Menaa , Paolo Giunio Pisano , Christian Theis, Applied Radiation and Isotopes 196 (2023) 110782

2.1.33 Eu-154

Property	Eu-154
Measurement method	Gamma spectrometry
Property info	Eu-152, Eu-154 and Eu-155 are produced by neutron capture in Eu-151 (47.8%) and Eu-153 (52.2%). Other routes to the production of Eu isotopes occur because of chain absorptions in Sm. Eu-152 decays (half-life: 13.5 y) by β– emission (maximum energy: 1.477 MeV) to Gd-152, which decays (half-life: 1.1×10^{14} y) by α emission. Eu-154 decays (half-life: 8.6 y) by β– emission (maximum energy: 1.85 MeV) to stable Gd-154. Eu-155 decays (half-life: 4.76 y) by β– emission (maximum energy: 2.52 keV) to stable Gd-155. Eu-152 and Eu-154 are the two dominant europium activation products in bioshield concrete and core graphite on a timescale of 10–20 years. Both have very large neutron capture cross-sections; Eu-152 is produced primarily by thermal neutrons, whereas Eu-154 also has a substantial resonance integral. The activities of Eu isotopes require consideration, owing to the presence of trace quantities of rare earth elements in source materials used in reactor graphite and bioshield concretes. Typically, sub-ppm levels of Eu and Sm parents generate sufficient activity on neutron activation to warrant precautionary measures during the early decommissioning of concrete bioshields. These Eu isotopes are measured by γ spectrometry at multiple energies or by β counting.
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	11.8 [4.1–23.4] mBq/g
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994). • Radiological characterisation for the clearance of burnable waste produced at CERN Richard Harbron, Renaud Charousset , Gerald Dumont , Matteo Magistris , Nabil Menaa , Paolo Giunio Pisano , Christian Theis, Applied Radiation and Isotopes 196 (2023) 110782

2.1.34 Eu-155

Property	Eu-155
Measurement method	Gamma spectrometry
Property info	Eu-152, Eu-154 and Eu-155 are produced by neutron capture in Eu-151 (47.8%) and Eu-153 (52.2%). Other routes to the production of Eu isotopes occur because of chain absorptions in Sm. Eu-152 decays (half-life: 13.5 y) by β– emission (maximum energy: 1.477 MeV) to Gd-152, which decays (half-life: 1.1 × 10^{14} y) by α emission. Eu-154 decays (half-life: 8.6 y) by β– emission (maximum energy: 1.85 MeV) to stable Gd-154. Eu-155 decays (half-life: 4.76 y) by β– emission (maximum energy: 2.52 keV) to stable Gd-155. Eu-152 and Eu-154 are the two dominant europium activation products in bioshield concrete and core graphite on a timescale of 10–20 years. Both have very large neutron capture cross-sections; Eu-152 is produced primarily by thermal neutrons, whereas Eu-154 also has a substantial resonance integral. The activities of Eu isotopes require consideration, owing to the presence of trace quantities of rare earth elements in source materials used in reactor graphite and bioshield concretes. Typically, sub-ppm levels of Eu and Sm parents generate sufficient activity on neutron activation to warrant precautionary measures during the early decommissioning of concrete bioshields. These Eu isotopes are measured by γ spectrometry at multiple energies or by β counting.
Technical info, interferences	-
Sample preparation	Can be directly measured by gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	?
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994).
Property	Radiation resistance
Measurement method	-
Property info	Waste forms containing significant amounts of radionuclides such as short-lived intermediate level waste integrate absorbed dose with time. High absorbed dose e.g., tens or hundreds of kGy may damage waste form: polymer, bitumen or concrete matrix. Thus, radiation resistance of waste form is usually, at least at laboratory scale, characterized. This characterization might also apply to the waste package envelope.
Technical info, interferences	-
Sample preparation	-
Chemical yield evaluation	-
MDA/Detection limit	-
Sources - detailed information	-

2.1.35 Na-22

Property	Na-22
Measurement method	Gamma spectrometry
Property info	This radionuclide is produced by the fast neutron Na-23(n,2n)Na-22 and the Na-23(γ,n)Na-22 reactions in the 100% abundant isotope Na-23. Na-22 decays (half-life: 2.6 y) by β+ and γ emission (maximum energy: 1.275 MeV) to Ne-22. Na-22 requires consideration during the decommissioning of fast reactors for the handling and disposal of sodium. Na-22 can be directly measured by γ spectrometry.
Technical info, interferences	-
Sample preparation	Na-22 can be directly measured by γ spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	6.9 [1.4–18.0] mBq/g
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994). • Jiaying Wang, Yu Chen, Zhongjian Ma, Lu Zhang, Zhen Zhang, Analysis of gamma-emitting radionuclides in soils around high energy accelerators, Radiation Medicine and Protection, Volume 3, Issue 4, 2022, Pages 171-174, ISSN 2666-5557 • Radiological characterisation for the clearance of burnable waste produced at CERN Richard Harbron, Renaud Charousset , Gerald Dumont , Matteo Magistris , Nabil Menaa , Paolo Giunio Pisano , Christian Theis, Applied Radiation and Isotopes 196 (2023) 110782

2.1.36 Ar-39

Property	Ar-39
Measurement method	Proportional beta counting LSC
Property info	This radionuclide is produced principally by the reaction K-39(n,p)Ar-39 with a cross-section of about 0.1 b. It may also be produced by neutron capture in naturally occurring Ar-38 via the reaction Ar-38(n,γ)Ar-39 with a cross-section of 0.8 b. Ar-39 decays (half-life: 269 y) by β- emission (maximum energy: 565 keV). K-39 is 93.3% abundant in natural potassium which is present, as a trace element, mainly in concrete at levels of thousands of ppm and in stainless steel and carbon steel at levels of hundreds of ppm. Ar-39 will begin to be a significant radionuclide several decades after shutdown. In reactor designs which make use of argon for inert gas blankets or air, significant production of Ar-39 will result. Examples include some fast breeder reactor (FBR) designs, the NRU reactor in Chalk River, and the Super Phenix. Ar-39 can be directly measured by proportional β- counting or liquid scintillation.
Technical info, interferences	-
Sample preparation	Ar-39 can be directly measured by proportional β- counting or liquid scintillation.
Chemical yield evaluation	-
MDA/Detection limit	Information needed
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994).

2.1.37 Zn-65

Property	Zn-65
Measurement method	Gamma spectrometry
Property info	This radionuclide is produced by the Zn-64(n,γ)Zn-65 reaction in the 49% abundant stable isotope; it decays (half-life: 244 d) via electron capture and β+ emission (maximum energy: 329 keV) to stable Cu-65. It appeared in significant concentrations in early boiling water reactor (BWR) units which employed heat exchangers containing Admiralty (29% Zn) or Muntz metal (40% Zn). In some cases, the condensed secondary steam was contaminated with stable zinc and was used for primary water make-up. The zinc contamination was brought into the primary reactor system where the stable zinc became neutron activated. It is measured by γ spectrometry at 1.112 MeV (annihilation energy).
Technical info, interferences	-
Sample preparation	The activity determination of gamma emitters from dissolved/mineralized samples of waste can be carried out using high resolution gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	17.5 [3.6–44.3] mBq/g
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994). • Radiological characterisation for the clearance of burnable waste produced at CERN Richard Harbron, Renaud Charousset , Gerald Dumont , Matteo Magistris , Nabil Menaa , Paolo Giunio Pisano , Christian Theis, Applied Radiation and Isotopes 196 (2023) 110782

2.1.38 Ba-133

Property	Ba-133
Measurement method	Gamma spectrometry
Property info	This radionuclide is produced by the Ba-132(n,γ)Ba-133 reaction in the 0.097% abundant isotope. Ba-133 decays (half-life: 10.5 a) by electron capture and by γ emission to the stable Cs-133 isotope. The main γ emitters, in decreasing order of radiation intensity, are 356, 303, 383 and 276 keV. Ba-133 is mainly found in the heavy concrete of biological shields based on the use of BaSO4 to increase the density. The Ba content in such concrete can amount to 30–40% by weight. The main γ emitter in concrete will then be Ba-133, which can be directly measured by γ spectrometry.
Technical info, interferences	-
Sample preparation	Can be directly measured by gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	8.0 [2.2–18.1] mBq/g
Sources - detailed information	• UNITED STATES DEPARTMENT OF ENERGY, Decommissioning Handbook, Rep. DOE/EM-0142P, DOE, Washington, DC (1994). • Radiological characterisation for the clearance of burnable waste produced at CERN Richard Harbron, Renaud Charousset , Gerald Dumont , Matteo Magistris , Nabil Menaa , Paolo Giunio Pisano , Christian Theis, Applied Radiation and Isotopes 196 (2023) 110782

2.1.39 Ho-166m

Property	Ho-166m
Measurement method	Gamma spectrometry
Property info	This radionuclide is produced by the Ho-165(n,γ)Ho-166m reaction in the 100% abundant stable isotope with a cross-section of 66.5 b. Ho-165 is present at ppb levels in steels and at ppm levels in concretes. Ho-166m decays (half-life: 1200 y) by β– emission (maximum energy: 1.314 MeV). The main γ rays, in decreasing order of radiation intensity, are at 184, 810 and 712 keV. Ho-166m is a contributor to the long term γ dose rate principally from graphite and has been identified in long deferment studies (>100 a) on GCRs. Sensitivity studies have indicated that the activation of parent Ho-165 in graphite will contribute about one half of the remaining γ dose rate at 135 a after shutdown. Ho-166m can be measured via chemical separation and γ spectrometry.
Technical info, interferences	-
Sample preparation	Can be directly measured by gamma spectrometry.
Chemical yield evaluation	-
MDA/Detection limit	Information needed
Sources - detailed information	• FELSTEAD, L.D., WOOLLAM, P.B., An Assessment of all Known Isotopes to Determine which might be Important in the Decommissioning of Thermal Reactors, Rep. TPRD/B/0386/N84, Central Electricity Generating Board Report, CEGB, London (1984).

2.1.40 Fissile material content

Property	Fissile material content
Measurement method	Active neutron interrogation (Differential die away (DDA))
Property info	Direct measure of fissile isotopes (total fissile mass), the quantity of most interest for nuclear criticality safety measurements.
Technical info, interferences	A pulsed electric neutron generator is used to induce fission in the material under assay. In between these pulses, detectors measure the distinctive time profile of emitted prompt neutrons, which is compared to the background response of the system. The time profile can indicate which material may be present and the integration of neutron counts (background subtracted) is proportional to the fissile mass present. Useful for U-235 and Pu-239 measurements (“total fissile”) in mixed streams.
Sample preparation	-
Chemical yield evaluation	-
MDA/Detection limit	The lower LoD is inversely proportional to the interrogating neutron flux.
Sources - detailed information	• Solid Radioactive Waste Characterisation Good Practice Guide, Jacobs, March 2022

2.1.41 Surface dose rate

Property	Surface dose rate
Measurement method	Health physics probes, typically scintillation- or GM- based detectors for the measurement of gross gamma dose rate.
Property info	A dose rate measurement is a simple, straightforward and very fast technique to assess the radiation level in Sv/h. Since it detects a total gamma signal, and not quantifying individual radionuclides, the measurement technique is useful as characterization method when a very stable nuclide vector is available.
Technical info, interferences	Sensitive to naturally occurring gamma radiation and other background gamma signals (no background discrimination). Potential variation in detector polar response requires consistent probe positioning. No energy information. Cannot determine individual radionuclide content. Energy-dependent response can be compensated for by appropriate design. Collimation of dose rate measurements can be beneficial for directional dose assessments, especially in situations where you want to determine the contribution of radiation from a specific direction or to minimize interference from surrounding sources. If the waste comes from a stable waste stream, the geometry is the same, the density distribution is homogeneous (waste packages are filled to a consistent density and height), then in special cases dose rate measurements can be used for isotope selective measurements (e.g. nuclear power plant ion exchange resins under stable reactor condition/reactor parameters. The conversion of dose to activity content is very dependent on the fingerprint (ETM, gamma emitting radionuclide composition). Not appropriate for waste streams varying in isotopic fingerprint or filling of waste packages.
Sample preparation	-
Chemical yield evaluation	-
MDA/Detection limit	LoD (Limit of Detection): instrument, background and contaminant specific. For monitoring of a 200 L drum filled with materials with a density of 1 g cm -3 and a net contact dose rate of 1 μSv/h derived activity is of the order of typically a few MBq. Very fast process. These types of probes are good for multiple, quick, measurements. Potentially can be used in conjunction with scaling factors.
Sources - detailed information	• Solid Radioactive Waste Characterisation Good Practice Guide, Jacobs, March 2022

2.1.42 Surface contamination

Property	Surface contamination
Measurement method	Hand-held alpha and beta contamination probes (often with gamma detection)
Property info	Monitoring of surface contaminated objects.
Technical info, interferences	Uncertainties: • Large, depending on the condition of the surface to be monitored. • Detection of alpha / beta from a surface is limited by overlaying absorbers e.g., oils, paint, rust.
Sample preparation	-
Chemical yield evaluation	-
MDA/Detection limit	LoD: instrument, background and contaminant specific. For DP6, typical alpha response: 18 cps per Bq/cm2. Typical beta response (Sr-90 / Y-90): 20 cps per Bq/cm2.
Sources - detailed information	• Solid Radioactive Waste Characterisation Good Practice Guide, Jacobs, March 2022

2.2 NDA Methods

2.2.1 Cavity Ring Down Spectroscopy

Measurement method	Cavity Ring Down Spectroscopy
Short	CRDS
Active/Passive	A
Description	The CRDS can provide the sensitivity required to detect fugitive radioactive emissions from nuclear waste outgassing and in the discharge air of nuclear power plants. In CRDS, the gas sample is placed inside an external resonant optical cavity consisting of high-reflectivity mirrors with an effective path length of several kilometres. A laser beam is coupled into the cavity, and the light exiting the cavity is focussed onto a detector. The laser coupling is suddenly switched off and the light decaying out of the cavity recorded. The signal is fitted with an exponential curve and the decay time, also called ring- down time (τ) determined. Additional losses due to analyte absorption result in a shorter ring-down time. Spectra are recorded at low pressure (~ 10 mbar) such that the absorption features are narrow and the peaks of the different molecules and isotopologues are well separated.
Complexity	Complex instrumentation.
Equipment cost	~100k€
Ease of deployment	High sensitivity Simplicity of use Low analysis cost
Training levels required to implement technique	#N/A - Collecting information
Performance guidance	CRDS technology is highly sensitive, capable of detecting trace levels of gases and isotopes at parts-per-billion (ppb) or parts-per-trillion (ppt) concentrations. It is also highly selective, allowing the measurement of specific gas species and isotopes in a complex gas mixture.

Considerations and Limitations	High sensitivity due to the multipass nature (i.e. long pathlength) of the detection cell. Immunity to shot variations in laser intensity due to the measurement of a rate constant. Wide range of use for a given set of mirrors; typically, ±5% of the center wavelength. High throughput, individual ring down events occur on the millisecond time scale. No need for a fluorophore, which makes it more attractive than laser-induced fluorescence (LIF) or resonance-enhanced multiphoton ionization (REMPI) for some (e.g. rapidly predissociating) systems. Commercial systems available. Spectra cannot be acquired quickly due to the monochromatic laser source which is used. Analytes are limited both by the availability of tuneable laser light at the appropriate wavelength and also the availability of high reflectance mirrors at those wavelengths. Expense: the requirement for laser systems and high reflectivity mirrors often makes CRDS orders of magnitude more expensive than some alternative spectroscopic techniques.
Applications	Used to detect fugitive radioactive emissions from nuclear waste outgassing and in the discharge air of nuclear power plants.

2.2.2 Cerenkov (Cherenkov) counting

Measurement method	Cerenkov (Cherenkov) counting
Short	Cerenkov (Cherenkov) counting
Active/Passive	A
Description	Cerenkov radiation results from a charged particle traversing a light transparent polar medium (e.g., water) with a velocity being higher than the phase velocity of light in this medium. This causes local electronic polarization of the dielectric molecules, which release electromagnetic radiation when returning to the ground state. For β-emitters in aqueous solution, a minimum energy of 262 keV is necessary. A reasonable efficiency is accessible for β-maximum energies exceeding 1 MeV. Cerenkov radiation does not require a scintillator. It can be detected in any medium (acidic or alkaline) and is not subject to chemical quenching.
Complexity	#N/A - Collecting information
Equipment cost	#N/A - Collecting information
Ease of deployment	#N/A - Collecting information
Training levels required to implement technique	#N/A - Collecting information
Performance guidance	#N/A - Collecting information
Considerations and Limitations	#N/A - Collecting information
Applications	#N/A - Collecting information

2.2.3 Determination of radiation resistance

Measurement method	Determination of radiation resistance
Short	-
Active/Passive	A
Description	#N/A - Collecting information
Complexity	#N/A - Collecting information
Equipment cost	#N/A - Collecting information
Ease of deployment	#N/A - Collecting information
Training levels required to implement technique	#N/A - Collecting information
Performance guidance	#N/A - Collecting information
Considerations and Limitations	#N/A - Collecting information
Applications	#N/A - Collecting information

2.2.4 Gamma Ray Spectrometry

Measurement method	Gamma Ray Spectrometry
Short	GRS
Active/Passive	P
Description	Gamma spectrometry is a routinely used non-destructive method enabling the identification and quantification of various gamma-emitting radionuclides in a wide range of sample types, including radioactive waste packages. By measuring the energies and intensities of gamma rays emitted by a sample, gamma ray spectrometry provides a spectrum revealing radionuclides and their abundance expressed in activity concentration. It is also used in destructive assay when the isotope of interest is present at low activity concentrations in a sample matrix with a high activity concentration. In the latter case, separation of the isotope of interest is then necessary prior to gamma spectrometric measurement, due to the Compton background caused by the high activity matrix. Energy calibration of the detector with known sources is needed before identification of unknown sources. In case of source activity estimation, information about detection efficiency is needed. Ratios between different measured gamma-ray peaks can also be used in the analysis of gamma-ray spectra. Attenuation from matrix needs to be taken into account especially for measurement of large items.
Complexity	Its complexity lies in the careful calibration, measurement setup, data analysis and interpretation needed to achieve accurate and meaningful results. Interpretation requires qualified staff.
Equipment cost	Up to > 100 k€ depending on flexibility and the spectral resolution required.
Ease of deployment	Influenced by whether the equipment is portable or fixed. Portable equipment (transportable detectors) offers greater flexibility and ease of deployment. Fixed installations may require more extensive setup and infrastructure.
Training levels required to implement technique	Skilled operators, proper training and attention to detail are essential to navigate the complexities and obtain reliable results.
Performance guidance	Efficiency calibration and energy calibration is needed. Determining detector efficiency for different gamma ray energies and geometries. Shielding and minimizing interference from ambient radiation sources.

Considerations and Limitations	Accurate calibration using standard radioactive sources or validated mathematical efficiency calculations are essential for obtaining meaningful results. Proper energy calibration ensures that the detected gamma ray energies correspond to the actual energies of the emitted radiation. The detector efficiency varies with gamma ray energy and geometry. Background radiation need to be accounted for. To address overlapping gamma ray energies from different radionuclides, complicating peak identification and quantification, high-resolution detectors are used. Low energy gamma rays may be challenging to detect due to background interference and Compton background and due to the absorption in the detector window High-precision measurements may require longer acquisition times to achieve adequate counting statistics.
Applications	Identification and quantification of gamma emitting radionuclide composition.

2.2.5 Neutron Activation Analysis

Measurement method	Neutron Activation Analysis
Short	NAA
Active/Passive	A
Description	NAA (Neutron activation analysis) is a nuclear analytical technique used to determine the composition of a sample. It is based on the principle of neutron activation, where a sample is exposed to a source of thermal or epithermal neutrons. The neutrons interact with the nuclei of the elements in the sample, causing some of them to become radioactive isotopes. After a suitable decay period, the radioactive isotopes are measured using a suitable detection technique, such as gamma-ray spectrometry. The gamma rays emitted by the radioactive isotopes are characteristic of the elements in the sample, and their energies and intensities can be used to identify and quantify the elements.
Complexity	Complex instrumentation.
Equipment cost	High.
Ease of deployment	Installed system.
Training levels required to implement technique	Higher burden of operator training. Understanding of neutron transport and potential interference is desirable.
Performance guidance	#N/A - Collecting information
Considerations and Limitations	#N/A - Collecting information
Applications	#N/A - Collecting information

2.2.6 X-ray Spectrometry

Measurement method	X-ray Spectrometry
Short	XRS
Active/Passive	A
Description	X-ray spectrometry is a technique used to analyse the elemental composition of a sample by measuring the characteristic X-ray emissions that are produced when the sample is irradiated with X-rays. These emissions are unique to each element and can be used to identify the elements present in the sample and determine their relative abundance. X-ray spectrometry can be used in radioactive waste characterization to identify and quantify the radioactive isotopes present in the waste.
Complexity	#N/A - Collecting information
Equipment cost	Of order €20,000.
Ease of deployment	#N/A - Collecting information
Training levels required to implement technique	#N/A - Collecting information
Performance guidance	#N/A - Collecting information
Considerations and Limitations	#N/A - Collecting information
Applications	#N/A - Collecting information

2.2.7 Hand-held alpha and beta contamination probes

Measurement method	Hand-held alpha and beta contamination probes
Short	-
Active/Passive	P
Description	Health physics probes, typically scintillation- or GM- based detectors for the detection of alpha and beta emissions from contaminated surfaces.
Complexity	Simple hand-held detector and dose rate meter combination.
Equipment cost	Of order €1,000.
Ease of deployment	Standard instrument used by Health Physics staff.
Training levels required to implement technique	Basic understanding of measurement process and suitability of instrument. Use of instrument regarding speed and distance of measurement from contaminated surface.
Performance guidance	LoD: instrument, background and contaminant specific. Very fast process. Potentially can be used in conjunction with scaling factors. Energy dependent response. For DP6, typical alpha response: 18 cps per Bq/cm2. Typical beta response (Sr-90 / Y-90): 20 cps per Bq/cm2. Uncertainties: • Large, dependant on the condition of the surface being monitored. • Detection of alpha / beta from a surface is limited by overlaying absorbers e.g., oils, paint, rust.
Considerations and Limitations	Sensitivity to gamma fields and interfering radiations. Cross talk for dual probes. Measurement in electromagnetic field for photomultiplier-based instruments. Some protection provided by mu-metal casing of photomultiplier tubes. Undiscovered light leaks / foil punctures / cable breaks can cause incorrect readings. The use of mismatched probe and ratemeter may also result in incorrect readings.
Applications	Monitoring of surface contaminated objects.

2.2.8 Gross gamma dose rate

Measurement method	Gross gamma dose rate
Short	-
Active/Passive	P
Description	Health physics probes, typically scintillation- or GM- based detectors for the measurement of gross gamma dose rate.
Complexity	Simple hand-held detector and ratemeter combination in a single unit.
Equipment cost	Of order €1,000.
Ease of deployment	Standard instrument used by Health Physics staff.
Training levels required to implement technique	Basic understanding of measurement process and suitability of instrument. Use of instrument regarding speed and distance of measurement from contaminated surface or from packaged material.
Performance guidance	LoD: instrument, background and contaminant specific. For monitoring of a 200 L drum filled with materials with a density of 1 g cm-3 and a net contact dose rate of 1 μSv/h derived activity is of the order of typically a few MBq. Very fast process. These types of probes are good for multiple, quick, measurements. Potentially can be used in conjunction with scaling factors.
Considerations and Limitations	Sensitive to gamma background signal. Energy-dependant response can be compensated for by appropriate design. Can be collimated. Sometimes this is beneficial for directional dose assessments. Waste packages to be filled to a consistent density and depth. Potential variation in detector polar response requires consistent probe positioning. No energy information. Cannot determine individual radionuclide content. The conversion of dose to activity content is very dependent on the fingerprint. Not appropriate for waste streams varying in isotopic fingerprint or filling of waste packages.
Applications	Monitoring of standard containers, uniformly filled with a non-varying gamma emitting content. Can also be used for monitoring of SCO Area survey monitoring.

2.2.9 Gross gamma counting systems

Measurement method	Gross gamma counting systems
Short	-
Active/Passive	P
Description	The total gamma signal is measured by plastic scintillator-based detectors arranged in a (configuration approaching) 4π geometry surrounding the item to be monitored.
Complexity	Will require calibration to obtain Bq/g value. Weighting scales and shielding may be included. Relatively straightforward to operate: Signal indicating OK / Not OK.
Equipment cost	Of order €10,000. Depending on the dimensions of the standard waste packages to be measured -> different sizes going from really small objects to boxes up to $1m^3$
Ease of deployment	Straightforward installation, but dependent on size of the monitors. Monitors for boxes up to $1m^3$ require technical engineers for setup and calibration, location is dependent on application but typically next to exit monitors. Requires electrical services and possibly communication with a remote office.
Training levels required to implement technique	Straightforward to use. More complex demand when setting to work.
Performance guidance	Very fast process. Potentially can be used in conjunction with scaling factors. Can use large scintillation detectors in near 4 π geometry. Increased sensitivity. Minimum Detectable Activity (MDA) of the order of 100 Bq. As with other techniques, MDA will be dependent on the instrument response characteristics and nature of the object being monitored.
Considerations and Limitations	No energy information available. Cannot determine individual radionuclide content. Fixed fingerprint required per waste stream. Sensitive to varying background.
Applications	Smaller versions usually applied for checking for presence of gamma emitters at controlled area boundaries. Larger models incorporating weighting scales for monitoring of standard waste packages.

2.2.10 Low resolution gamma spectroscopy

Measurement method	Low resolution gamma spectroscopy
Short	LRGS
Active/Passive	P
Description	LRGS involves the analysis of gamma radiation emitted by radioactive materials using a system with limited ability to distinguish between different energy levels of gamma rays. This technique provides no fine energy discrimination, so no detailed information of specific radionuclides, but the technique is very useful for fast screening and indication of the presence of radioactive elements. Typically, Sodium Iodide (NaI(Tl)) scintillator optically coupled to a photomultiplier in a metal casing. Requires High Voltage (HV), Analogue to Digital Converter (ADC) and Multichannel Analyser (MCA) electronics and software. These functions are commonly incorporated into a simple “plug and play” unit. Other scintillating materials available, e.g., Bismuth Germanate (BGO), Caesium Iodide (CsI).
Complexity	More complex than gross gamma-ray dose meters, but relatively straightforward to use. “Point and shoot “capability. Data logging capability. Requires HV, ADC and MCA electronics and software. Now available as a single “plug and play” unit.
Equipment cost	Of order €10,000. This is applicable to individual detectors but complex NaI systems can cost substantially more
Ease of deployment	Straightforward deployment. No cooling requirement. Low maintenance. Hand held battery powered models available. Automated spectral analysis against stored data libraries. Can store spectra for later analysis.
Training levels required to implement technique	Operator training more complex as training on setting up and analysis of spectra required.
Performance guidance	LoD: instrument, background, source and contaminant specific. Uncertainties: • Variation in proportion of gamma emitters in a waste stream can be detected and accounted for. • Energy resolution ~7% at 661 keV for NaI. • An efficiency calibration should be performed for each geometry. • If properly calibrated, can correct for variation in density of waste package content. • Large crystal volumes available with large detectors can be very sensitive, much more than dose rate measurement. • Specially shaped crystals can be manufactured.

Considerations and Limitations	Available in a range of crystal sizes from ~1.5 cm3 to ~4000 cm3. This means the detectors are good for non-man access surveys that need to fit through small apertures, or for assaying wide areas due to the high absolute detection efficiency of a large crystal. Limited resolution limits technique to simple spectra. Spectrum quality can be quite dependent on the total spectrum count rate. This may manifest in various ways such as peak shifting and shape changing. Hence, complex spectra may be incorrectly interpreted. Crystals and photomultiplier tubes are fragile. Susceptible to electrical noise, magnetic fields and thermal drift.
Applications	Assay of gamma-emitting waste streams and packages.

2.2.11 Intermediate resolution gamma spectroscopy

Measurement method	Intermediate resolution gamma spectroscopy
Short	IRGS
Active/Passive	P
Description	Cadmium Zinc Telluride (CZT) semiconductor detector coupled with HV, ADC and MCA electronics and software. Scintillation detectors using $LaBr_3$ are also available.
Complexity	Similar to low resolution gamma spectroscopy. Detector, HV ADC and MCA electronics available in a single unit.
Equipment cost	Of order €10,000.
Ease of deployment	Straightforward deployment. No cooling requirement. Low maintenance. Hand-held battery powered models available. Automated spectral analysis against stored data libraries. Can store spectra for later analysis.
Training levels required to implement technique	Operator training more complex as training on setting up and analysis of spectra required.
Performance guidance	LoD: instrument, background, source and contaminant specific. Uncertainties: • Energy resolution ~2% at 661 keV. • Variation in proportion of gamma emitters in a waste stream can detected and accounted for. • An efficiency calibration should be performed for each geometry. • If properly calibrated can correct for variation in density of waste package content. • Can be used in higher dose rate environments subject to crystal size. • Small size of unit makes the equipment more amenable to specialist monitoring e.g. mounting on ROV, underwater submersibles and airborne drones.
Considerations and Limitations	Small crystal size (maximum ~1.5 cm3 for CZT) limits response to higher energy photons. Small crystal size requires higher photon flux to obtain a good spectrum in a reasonable time. Peak asymmetry in CZT spectra is observed and needs to be taken into account during analysis. $LaBr_3$ detectors are available with size up to 3x3 inch.
Applications	Assay of gamma-emitting waste streams and packages.

2.2.12 High resolution gamma spectroscopy

Measurement method	High resolution gamma spectroscopy
Short	HRGS
Active/Passive	P
Description	High Purity Germanium (HPGe) semiconductor detector coupled with HV, ADC and MCA electronics and software.
Complexity	Complex demand for operation and analysis of spectra (in case of electrically cooled operation is not so much different than other gamma spectrometers, more complex in case of non-routine measurements. Higher energy resolution enables the analysis of more complex spectra. Detector, HV ADC and MCA electronics available in a single unit. Requires cooling (Liquid nitrogen or electrical cooling available).
Equipment cost	Of order €50,000 to €250,000.
Ease of deployment	Installed systems, transportable or Portable Hand-held Battery powered models available. Automated spectral analysis against stored data libraries. Complex peak search and deconvolution for analysis of acquired spectra. Efficiency curves required for each measurement geometry.
Training levels required to implement technique	Operator training complex as training on setting up, function checking and maintenance. Analysis of complex spectra requires an experienced operator.
Performance guidance	LoD: instrument, background, source and contaminant specific. Uncertainties: • Energy resolution ~0.2% at 661 keV. • Complex spectra with multiline emission can be analysed. • An efficiency calibration should be performed for each geometry. Can generate efficiency calibration using mathematical methods. Off the shelf energy calibration software available. • If properly calibrated can correct for variation in density of waste package content.
Considerations and Limitations	Cost. Requires an experienced operator for non-routine use and interpretation of acquired spectra. Requires detailed instructions for the operation of the High Resolution Gamma Spectrometer and for use of software. Maintenance burden. Complex spectra are open to misinterpretation. Cryogenic cooling requirement. Sensitivity vs. dead time issues.
Applications	Assay of gamma-emitting waste streams and packages. Range of crystal volumes and shapes (cylindrical, planar, well type) available. Installed systems can be set up for automated gamma assay of standard waste items, e.g. 200 L drums.

2.2.13 High resolution gamma spectroscopy segmented or tomographic gamma scanners

Measurement method	High resolution gamma spectroscopy segmented or tomographic gamma scanners
Short	HRGS-SGS/TGS
Active/Passive	A/P
Description	HPGe solid state detector coupled with HV, ADC and MCA electronics and software, radioactive sources for transmission measurements and mechanical interface with drum rotation.
Complexity	Highly complex. Detector system linked with collimated source and drum rotation and / or detector deployment.
Equipment cost	Of order €500,000 -1,000,000.
Ease of deployment	Installed system.
Training levels required to implement technique	High degree of Operator training as training on setting up, function checking and maintenance. Analysis and interpretation of multiple complex spectra requires an experienced operator.
Performance guidance	Used to correct for heterogeneity by taking measurements over a number of positions. Use of a transmission source to correct for density. Systems require extensive calibration, setup and commissioning effort.
Considerations and Limitations	Cost. Complexity. Maintenance burden. Complex spectra are open to misinterpretation. Reliability of mechanical interfaces. The design of these systems need to take into account source management, operator dose uptake and shielding issues.
Applications	Installed systems set up for automated gamma assay of standard waste items, e.g. 200 L drums. Tomographic gamma scanning is potentially the most accurate technique as it corrects in three dimensions.

2.2.14 Gamma imaging

Measurement method	Gamma imaging
Short	-
Active/Passive	P
Description	The device combines an optical camera with a gamma detector to overlay radiation hotspots on top of an image to highlight the source of gamma emissions in the field of view, usually as a false colour overlay. Three basic types of gamma imager are routinely used: • Scanning Collimator. A highly collimated detector scans over the predetermined field of view and records the gamma response of the detector. • Coded Aperture. A complex shaped mask of shielding material casts a (gamma) shadow in a position-sensitive detector. The shape of the shadow can be used to determine the position of sources of activity. • Pinhole Camera. A small aperture through a dense shielding material (typically tungsten) allows gamma rays to be projected onto a position sensitive detector array. The principle of operation is similar to an optical pinhole camera where an inverted image of a scene is projected onto a photographic film.
Complexity	Straightforward to use.
Equipment cost	Of order €100,000 - €200,000.
Ease of deployment	Straightforward to set up.
Training levels required to implement technique	Understanding of scanning speeds, extent of scanning field and effect of angular resolution on final image desirable. Interpretation of image and quantification of activity content requires understanding of shielding.
Performance guidance	Require expert analysis for activity quantification assessments. Some devices can determine images at more than one energy simultaneously, e.g., Cs-137 and Co-60.
Considerations and Limitations	Cost. Scanning Collimator and Pinhole Camera types can be bulky / heavy due to use of a tungsten collimator. Can take significant time to survey an area, dependant on angular resolution and activity levels present. Coded Aperture types can be affected by background radiation, primarily radiation outside the field of view.
Applications	Typically, gamma imaging has been used for scanning of active facilities, plant and in-cell measurements for the identification of hot spots to inform decisions on decommissioning planning, waste characterisation and waste categorisation. Data can also be used for Health Physics ALARP planning.

2.2.15 Passive neutron coincidence counting

Measurement method	Passive neutron coincidence counting
Short	PNCC
Active/Passive	P
Description	Detection of co-incident neutron emissions due to spontaneous fission.
Complexity	Neutron detection and electronic timing circuitry to determine amount of fertile even-numbered actinides.
Equipment cost	Of order €1,000,000, applied to general waste packages (200l drum, 1 m3 concrete, etc.), depends on number and size of detectors, etc.
Ease of deployment	Installed system.
Training levels required to implement technique	Modest burden of operator training. Understanding of neutron transport and potential interference is required.
Performance guidance	Most often used to assess plutonium content by direct measurement of Pu-240 and inferred Pu-239 content by prior knowledge of the isotopic ratio. No knowledge of chemical composition required if composition of the standard is similar to the waste stream. MDA: mg Pu-240 equivalent.
Considerations and Limitations	Significant moderation in the waste matrix prior to reaching a detector, e.g. larger packages or presence of neutron absorbers, may severely limit method. (α, n) reactions can interfere if not properly rejected by the timing circuitry due to pile up of accidental coincidences. Potential interference from other spontaneous fission isotopes (e.g. Cf or Cm). Requires knowledge of isotopic composition. Requires knowledge of and correction for leakage multiplication. Sensitivity to changes in efficiency due to neutron absorbers / moderators or variation of the position of the plutonium within chamber if measurement chamber is not well designed.
Applications	Assay of even-numbered Pu isotopes in waste packages.

2.2.16 Active neutron coincidence counting

Measurement method	Active neutron coincidence counting
Short	ANCC
Active/Passive	A
Description	Use of an external neutron source to induce prompt fission in material under assay.
Complexity	Detection of prompt fission neutrons induced by an external random neutron source.
Equipment cost	Of order €1,500,000 – 2,000,000.
Ease of deployment	Installed system.
Training levels required to implement technique	Modest burden of operator training. Understanding of neutron transport and potential interference is desirable.
Performance guidance	Typically used for the assay of U-235 and Pu-239. Selection of lower energy neutron source can be used to minimise fissioning of U-238. Detection limits are typically at the gram level.
Considerations and Limitations	Lumps of fissile material can cause underestimation of the true fissile mass, due to self-shielding as a result of the low penetrability of interrogating thermal neutrons. Accidental coincidence rates may be high from installed neutron sources. Difficulty in obtaining purely random neutron sources. Raised dose to operators from constantly exposed neutron sources if design does not take this risk into account.
Applications	Typically used for the measurement of uranium product waste, or for the radiological characterisation of un-irradiated fuel elements or assemblies.

2.2.17 Active neutron interrogation (Cf shuffler)

Measurement method	Active neutron interrogation (Cf shuffler)
Short	-
Active/Passive	A
Description	An external Cf-252 neutron source is used to induce fission in the material under assay. Adjacent detectors measure the delayed neutron signal, with the response proportional to the amount of fissionable material present.
Complexity	Detection of delayed neutrons from the assay material following fission induced by repeated rapid introduction and withdrawal of external Cf-252 neutron source.
Equipment cost	Of order €2,000,000.
Ease of deployment	Installed system.
Training levels required to implement technique	Higher burden of operator training. Understanding of neutron transport and potential interference is desirable.
Performance guidance	Detection limits typically tens – hundreds of mg of fissile material. Used for measurement of Pu-239 and U-235. Large interrogation flux permits good penetration of matrix and measurement of matrix properties. Can be designed to measure a wide variety and size of container types and waste forms.
Considerations and Limitations	Potentially large matrix effects requiring a complex correction technique when applied to waste assay. Extensive calibration required, depending upon the application (usually requiring much longer measurement time than for a passive neutron system). Larger maintenance burden renewing the Cf-252 source.
Applications	Direct measure of fissile isotopes, but incapable of directly discriminating between them. Useful for U-235 and Pu-239 measurements (“total fissile”) in mixed streams. Widely used for measurements of “uranium–only” streams.

2.2.18 Active neutron interrogation (Differential die away (DDA))

Measurement method	Active neutron interrogation (Differential die away (DDA))
Short	0
Active/Passive	A
Description	A pulsed electric neutron generator is used to induce, fission in the material under assay. In between these pulses, detectors measure the distinctive time profile of emitted prompt neutrons, which is compared to the background response of the system. The time profile can indicate which material may be present and the integration of neutron counts (background subtracted) is proportional to the fissile mass present.
Complexity	Very significant maintenance burden.
Equipment cost	Of order €1,000,000
Ease of deployment	Installed system.
Training levels required to implement technique	Higher burden of operator training. Understanding of neutron transport and potential interference is desirable.
Performance guidance	Potentially extremely sensitive. Only suitable for non-moderating materials. Neutron interrogation is typically performed using high intensity 14 MeV neutrons from “D-T” pulsed neutron generators, but 2.5MeV neutrons from “D-D” generators can also be used. The lower LoD is inversely proportional to the interrogating neutron flux. DDA is also well-suited to high background assay applications (e.g. irradiated fuel with high curium content) that demand a high signal- to-background ratio. Milligram quantities of Pu-239 or U-235.
Considerations and Limitations	Only suitable for non-moderating materials. Very high maintenance burden. Potentially large matrix and lump absorption effects requiring a complex correction technique when applied to waste assay. Considerable initial characterisation using known masses of fissile material is required to understand the response of the system.
Applications	Direct measure of fissile isotopes (total fissile mass), the quantity of most interest for nuclear criticality safety measurements. Useful for U-235 and Pu-239 measurements (“total fissile”) in mixed streams.

2.2.19 Calorimetry

Measurement method	Calorimetry
Short	-
Active/Passive	P
Description	The measurement of heat generated by certain radioactive decay processes is used to quantify the amount of radioactivity
Complexity	Relatively simple technique (single chamber calorimeter) to somewhat more complex setups (*e.g.* differential heat-flow calorimeter).
Equipment cost	Of order €100,000, but depends strongly on the considered volume.
Ease of deployment	Installed system.
Training levels required to implement technique	Low operator training requirement.
Performance guidance	Calorimetry is potentially the most accurate non-destructive assay technique, given an isotopic composition. The sample's thermal power can be related directly to fundamental physical quantities (i.e., voltage and current) and is mostly immune to the matrix effects that affect neutron and gamma-ray techniques. Only if gamma decay represents a significant portion of the thermal power, part of the energy may be deposited outside of the measurement chamber, making the efficiency depend on the matrix properties. Detection limits can range from less than 1 μW for volumes smaller than one litre up to several mW when considering volumes of several hundreds of litres.
Considerations and Limitations	Requires knowledge of isotopic composition. Time necessary to achieve thermal equilibrium in the sample measurement chamber can go from less than one hour for small samples up to days or even weeks in case of large volume calorimetry with samples with large thermal capacity. Less portable than other NDA techniques. Not yet practically proven for routine bulk waste assay. Potential for interference from heat from chemical reaction.
Applications	It is mainly used for Pu, Am-241 and H-3, as these have high enough specific activities and corresponding thermal power to generate sufficient heat for most practical measurement purposes.

2.2.20 Laser-Induced Breakdown Spectroscopy

Measurement method	Laser-Induced Breakdown Spectroscopy
Short	LIBS
Active/Passive	A
Description	An energetic laser excites the surface and the resulting light emitted is analysed to identify elemental constituents
Complexity	Relatively straightforward method deployed in the field in non-nuclear sectors.
Equipment cost	Of order €100,000.
Ease of deployment	Straightforward. May be issues associated with the use of high-powered lasers. Hand-held variants commercially available.
Training levels required to implement technique	Straightforward. Laser safety issues to address.
Performance guidance	Potentially deployable for remote operation. Use of optical fibres to access difficult to reach locations. Narrow focus of laser requires rasterization of area to be analysed. Technique may not detect some low-Z elements.
Considerations and Limitations	Only measures the surface. Quantitative analysis difficult. Not strictly non-destructive analysis as a small amount of material is removed by the excitation process. Narrow focus of laser may result in inadequate sampling and misleading results for non- homogeneous materials, e.g., concrete, mixed waste.
Applications	Identification of the elemental composition of unknown waste.

2.2.21 Laser ablation mass spectroscopy

Measurement method	Laser ablation mass spectroscopy
Short	LAMS
Active/Passive	A
Description	Use of a high-power laser to ablate the surface under examination. The ablated material can then be collected for subsequent laboratory analysis or analysed in situ by injection into an Inductively Coupled Plasma Mass Spectrometry.
Complexity	Relatively new technique systems have been developed to be deployed in the field.
Equipment cost	Of order €100,000.
Ease of deployment	In development.
Training levels required to implement technique	In development.
Performance guidance	Potentially deployable for remote operation. Use of optical fibres to access difficult to reach locations. Can produce samples for subsequent analysis in situ by mass spectrometry.
Considerations and Limitations	Only measures the surface. Quantitative analysis difficult. Not strictly non-destructive analysis as a small amount of material is removed by the excitation process.
Applications	Identification of the elemental composition of unknown waste.

2.2.22 Portable X-ray Fluorescence (XRF)

Measurement method	Portable X-ray Fluorescence (XRF)
Short	XRF
Active/Passive	A
Description	XRF is a non-destructive technology for material analysis, by measuring the secondary fluorescent rays emitted from a sample when excited by an X-ray source. The method is employed for quantitative and qualitative analysis of material composition since it is able to identify the unique fingerprint of each element.
Complexity	Straightforward, result is immediately available to the operator. Hand-held XRF in common use in the metals and recycling industries.
Equipment cost	Of order €20,000.
Ease of deployment	Straightforward. Hand-held or portable XRF analyzers are designed to provide instant elemental analysis in situations where immediate feedback is necessary
Training levels required to implement technique	Straightforward. Users to be trained in radiation safety.
Performance guidance	Potentially deployable for remote operation. Principal use is for qualitative assay. Can be calibrated for quantitative assay. Potentially mountable on ROVs.
Considerations and Limitations	Use of ionising radiation (x-rays). May suffer from matrix effects, limiting the detection of trace constituents. No sample preparation is involved, although care may need to be taken during selection of the test area.
Applications	Identification of the elemental composition of material. Rapid determination between metal types and alloys.

2.2.23 Raman spectroscopy

Measurement method	Raman spectroscopy
Short	RS
Active/Passive	A
Description	Irradiation of the item (solid liquid or gas) by an appropriately tuned laser interacts with molecular vibrations, phonons or other excitations in the system, yielding information on the characterisation of the molecular / chemical nature of the material.
Complexity	Can be used to interrogate items at a distance. Portable devices available.
Equipment cost	Of order €20,000.
Ease of deployment	Straightforward. Portable devices available.
Training levels required to implement technique	Straightforward. Laser safety issues to address.
Performance guidance	Potentially deployable for remote operation. Use of fibre optic cable can permit remote operation of probe from laser source. Principal use is for qualitative assay.
Considerations and Limitations	Heating by absorption of laser may alter condition of sample being analysed.
Applications	Identification of chemical composition of materials under test.

2.2.24 Sludge yield strength

Measurement method	Sludge yield strength
Short	-
Active/Passive	A
Description	Pushing of an instrumented tip down into the sludge at a controlled rate (cone penetrometer) or rotation of a vane. Measurement of the torque or resistance on the tip using a load cell and can be equated to shear strength.
Complexity	Simple hand-held tools through to complex remote installations including hydraulic driven systems.
Equipment cost	Of order €20,000.
Ease of deployment	Very dependent on accessibility and hazard presented by tank / pond contents.
Training levels required to implement technique	Straightforward for manual techniques.
Performance guidance	Measurement of yield strength of sludges by deployment of vane or cone penetrometer. Should be able to measure over depth of sludge. Need to consider accessibility and distance from point of access (hatch / flange) to bottom of sludge.
Considerations and Limitations	Reproducibility questionable. Decontamination of retrieved equipment. Interpretation. May be significant apparent variation across tank / pond area. Requires access to sludge which may preclude manual methods due to chemical and radiation hazards. Vane penetrometer measurements disturbs sludge so continuous measurement through depth of sludge may not be possible and may require ROV deployment.
Applications	Provides information on the mobility of sludges. Input in the design of sludge transfer systems.

2.2.25 Sludge rheology

Measurement method	Sludge rheology
Short	-
Active/Passive	A
Description	Principally a laboratory-based method of measuring fluid flow properties. Portable and online viscometer systems commercially available.
Complexity	Simple hand-held tools through to complex remote installations including hydraulic driven systems.
Equipment cost	Of order €10,000.
Ease of deployment	Very dependent on accessibility and hazard presented by tank / pond contents.
Training levels required to implement technique	Training required to interpret results.
Performance guidance	Online systems implemented in the waste water industry for low viscosity fluids.
Considerations and Limitations	Calibration for absolute values may be difficult.
Applications	Provides information on the flow characteristics of sludges. Input in the design of sludge transfer systems.

2.2.26 Particle size distribution

Measurement method	Particle size distribution
Short	-
Active/Passive	A
Description	Measurement of scattered light of sample illuminated by laser.
Complexity	Standard industrial technique.
Equipment cost	Of order €10,000.
Ease of deployment	Straightforward but will be dependent on accessibility and hazard presented by tank / pond contents.
Training levels required to implement technique	Standard method, no special training required.
Performance guidance	Typical measurement range 0.1 µm to 1000 µm. On-line systems commercially available.
Considerations and Limitations	Calibration. Sampling in stratified system. Ambient light.
Applications	Real time measurement of particle size. Characterisation of waste stream for sludge / fluid transfer engineering design purposes.

2.2.27 Particle shape

Measurement method	Particle shape
Short	-
Active/Passive	A
Description	Currently a laboratory method for the offline the analysis of samples.
Complexity	Particle imaging with cameras and software for analysis of images.
Equipment cost	Of order €10,000.
Ease of deployment	Straightforward.
Training levels required to implement technique	Commercial systems stated to be easy to use.
Performance guidance	Typical measurement range <1 µm to >1000 µm dependant on optical system. Can be used for measurement of dry powders, particles in suspension or on filters.
Considerations and Limitations	Online / in situ systems limited to dry powders. Analytical software may be easy to use but can be computationally expensive and subject to errors, e.g., not identifying overlapping particles. Can be sensitive to image resolution.
Applications	Analysis of particle shape in samples.

2.2.28 Sonar, including ultrasound

Measurement method	Sonar, including ultrasound
Short	-
Active/Passive	A
Description	Ultrasonic sensors are commonly used for tank level measurement and control.
Complexity	Straightforward.
Equipment cost	Of order €1,000.
Ease of deployment	Standard industrial method.
Training levels required to implement technique	Standard method, no special training required.
Performance guidance	Typical Resolution of 2 mm. Typical Accuracy 0.25% of total measurement range.
Considerations and Limitations	Subject to interferences or spurious echoes if installed in high tank depths. Ability to detect interface of sub surface sludge layers is limited
Applications	Measurement of liquid depth in tanks (and hence volume).

2.2.29 Temperature

Measurement method	Temperature
Short	-
Active/Passive	P
Description	Measurement of temperature of objects (tanks, pipes, packages and waste items).
Complexity	Simple commercially available equipment.
Equipment cost	Of order €500.
Ease of deployment	Straightforward.
Training levels required to implement technique	None.
Performance guidance	Use of Infra-Red device enables the remote measurement of temperature. Use of in built laser to assist targeting. A larger distance to spot size ratio is desirable for a narrower field of view.
Considerations and Limitations	A wide field of view may cause misleading temperature reading. Maximum useable distance ~10 m (dependant on size of object and distance to spot size ratio). Covering of measured object may cause misleading readings.
Applications	Temperature measurement to distinguish physically hot vessels, process lines or items. Indicator of on-going chemical reactions in items. Indication that package contents is not stable.

2.2.30 Pressure

Measurement method	Pressure
Short	-
Active/Passive	P
Description	Measurement of the pressure of items.
Complexity	Simple commercially available pressure gauges.
Equipment cost	Of order <€1,000.
Ease of deployment	Straightforward.
Training levels required to implement technique	None.
Performance guidance	Expect waste content to be stabilised and conditioned prior to storage in waste package. Pressure measurement of packaged waste can indicate unstable waste content.
Considerations and Limitations	Requires penetration of any containment to enable measurement of internal pressure. Anticipation that pressure measurement is required. Requirement of installation of pressure valve prior to introduction of radioactive material. Storage in high dose rate area may prevent retrospective fitting of pressure connection.
Applications	Indicator of on-going chemical reactions causing gas production in items. Indication that package contents is not stable.

2.2.31 Laser scanning

Measurement method	Laser scanning
Short	-
Active/Passive	A
Description	Use of controlled steering of laser beams with a laser rangefinder to perform dimensionally accurate surveys. Handheld versions available.
Complexity	Common method commercially available.
Equipment cost	Of order €10,000.
Ease of deployment	Simple to setup.
Training levels required to implement technique	2-day commercial training courses available.
Performance guidance	Typical resolution of point data of 2 to 6 mm at 10 m depending on model. Weight up to 6 kg. Typical use of a Laser class 1 should not cause health and safety issues. May be used to show dimensional changes of packages.
Considerations and Limitations	Mountable on a remote-controlled arm. There is no need to be in physical contact with measured objects. 3D representation of surveyed space may aid interpretation Initial cost is high. Processing of large data files requires high computing capability Software may be complex.
Applications	Scanning to obtain dimensional survey information of the arrangement of waste vaults and contents. Scanning of area to obtain dimensional information on tanks, vessels, equipment and pipework to confirm design drawings prior to retrievals.

2.2.32 Visual inspection

Measurement method	Visual inspection
Short	-
Active/Passive	P
Description	Simple inspection of items either individual or in the presence of with other objects.
Complexity	Straightforward, for unaided viewing.
Equipment cost	No cost - but see Considerations and Limitations.
Ease of deployment	Simple - direct line of sight but see Considerations and Limitations.
Training levels required to implement technique	Training required to aid interpretation of viewed image.
Performance guidance	May require installation of light sources to see items. Can distinguish features by colour and visible surface texture. Can only view surface features. Will require additional equipment to view at wavelengths outside the visible spectrum, e.g. infra-red. May require installation of mechanisms to reduce dose to eye: Lead glass windows, mirrors, periscopes. Viewing through coloured filters may improve contrast.
Considerations and Limitations	Difficult to determine distance with unaided eye. Potential dose to eye may prevent direct viewing of item. May require installation of mechanisms to reduce dose to eye: Lead glass windows, mirrors, periscopes. Glare and reflections may obscure details Angular Resolution of the unaided eye (approximately 0.02° approx. 1mm at 3m) limits the detail that can be seen. Direct line of sight may require
Applications	Identification of individual items by shape, colour or texture. Visual inspection of packaged items. Visual inspection of sludge and resins. Staining may indicate leakage. Bulging of containers and drums may indicate on going reactions causing pressurisation.

2.2.33 Video scanning

Measurement method	Video scanning
Short	-
Active/Passive	P
Description	Use of video cameras to record images of scanned area.
Complexity	Common method commercially available. Combination of video camera or fiberscope and monitor to provide remote imaging capability.
Equipment cost	Of order €100 -€1,000's
Ease of deployment	Simple straight forward use. Issues may arise with accessing location to be inspected.
Training levels required to implement technique	As per visual inspection.
Performance guidance	Resolution. Imaging possible in visible and non-visible spectrum. Recording of image for record purposes. Stereoscopic imaging to aid quantification of size and shape. Use in combination with other types of instruments. Can be deployed and image otherwise non-viewable areas via extending rods or ROVs. Can be used in relatively high dose rate areas that preclude human presence. Consider use of wireless camera if deployed in contaminated area.
Considerations and Limitations	Radiation damage. Interpretation of image can be challenging especially if narrow viewing field. Umbilical between camera and monitor may become contaminated Light source required.
Applications	Identification of individual items by shape, colour or texture. Visual inspection of packaged items.

2.3 DA Methods

2.3.1 Alpha Spectrometry

Analytical technique	Alpha Spectrometry
Short	AS
Description	Alpha spectrometry is a type of radioactivity measurement technique used to determine the presence and quantity of alpha-emitting isotopes in a sample. Alpha particles are high-energy, heavy, and positively charged particles that are produced during the decay of certain isotopes. Alpha spectrometry is based on the principle of measuring the energy and number of alpha particles emitted by a sample. In alpha spectrometry, the sample is typically deposited on a thin foil or placed in a special chamber. The alpha particles emitted by the sample are detected by a suitable detector, such as a Passivated Implanted Planar Silicon Detector, and their energies are measured. The energy spectrum of the alpha particles provides information about the isotopes present in the sample, and the number of alpha particles detected can be used to quantify the amount of radioactivity.
Emission type	Alpha radiation
Detector	Semiconductor
Applicable determinants	Pu-238, Pu-239/240, Am-241, Cm-242, Cm-243/244, Th-228, Th-230, Th-232, U-234, U-235/236, U-238, Np-237, Ra-226, Po-210.
Advantages / Disadvantages	Advantage: Low minimum detectable activity. Disadvantages: Long count times (can be as high as days, depending on the instrument background and source activity). Requires complex radiochemistry to produce thin sources for counting. Cannot distinguish between some alpha- emitting radionuclides (e.g. Pu-239/Pu-240, U-235/U-236, U-232/U-233 and Cm-243/244. In these circumstances, it would be necessary to resort to a mass spectrometric technique Th-228 recoil contamination and ingrowth of Ra-226 daughter both have adverse impacts with regard to impact on instrument backgrounds.
Performance measures (LoD, uncertainties)	LoDs typically in the range 0.0001 to 0.005 Bq/g.
Pitfalls	-

2.3.2 Liquid Scintillation Counting (LSC)

Analytical technique	Liquid Scintillation Counting (LSC)
Short	LSC
Description	The basic principle of liquid scintillation counting is to dissolve the sample containing the pure beta or alpha emitting radionuclides in a solution called scintillation cocktail, which is a mixture of a solvent and a scintillation agent. The scintillation agent fluoresces (emits light) when it interacts with the radioactive particles present in the sample. This light emission is proportional to the number of radioactive decay events and can be measured using a photomultiplier tube or a scintillation counter. Liquid scintillation counting is particularly useful for measuring low-energy beta emitters and alpha emitters, which can be difficult to detect using other methods.
Emission type	Beta (also alpha) radiation
Detector	Photomultiplier
Applicable determinants	e.g. H-3, C-14, Sr-90, Pu-241, Fe-55, Ni-63, Tc-99, Cl-36, Sm-151, Pm-147, gross alpha and beta activities.
Advantages / Disadvantages	Lower spectrum resolution compared to alpha and gamma spectrometry. Limited use for nuclide identification. May require element to be radiochemically separated before measurement. Can be used for gross beta measurement to include low energy beta emitters.
Performance measures (LoD, uncertainties)	LoDs typically in the range 0.05 to 1 Bq/g.
Pitfalls	LoDs for Fe-55 and Ni-63 in steels / alloys can be limited by the stable element concentrations. Potential for quenching and chemiluminescence effects leading to erroneous results.

2.3.3 Gamma Ray Spectrometry

Analytical technique	Gamma Ray Spectrometry
Short	GRS
Description	Gamma spectrometry is a routinely used method in non-destructive assay of radioactive wastes, but it is also used in destructive assay when the isotope of interest is present at low activity concentrations in a sample matrix with a high activity concentration. Separation of the isotope of interest is then necessary prior to gamma spectrometric measurement, due to the Compton background caused by the high activity matrix.
Emission type	Gamma radiation
Detector	High purity germanium
Applicable determinants	e.g., Am-241, Co-60, Cs-137, Eu152, Eu-154, Eu-155, I-131, Np-237/Pa-233, Mn-54, Nb-94, Nb95, Ra-226, Ru-106, Sb-125, U235, Zn-65, Zr-95, multiple nuclides in the U, Th, Ac & Np decay series.
Advantages / Disadvantages	High Resolution. Wide range of radionuclides determined simultaneously from a single measurement. No complex radiochemistry required, only sample preparation / homogenisation. Relatively rapid.
Performance measures (LoD, uncertainties)	LoDs vary widely with gamma emission probability and energy / counting efficiency. Typically sub 0.005 Bq/g for Cs-137, Co-60 and Am-241.
Pitfalls	Sample heterogeneity, particularly where samples contain highly localised “hot spots”, or where the material is difficult to size-reduce. Measurement of an acid digest can overcome this, but LoDs may be compromised as a result. Interferences where peaks have similar energy (e.g., Ra-226 & U-235). LoD affected by overall activity of sample, e.g. high Cs-137 or Co-60 will raise LoDs for other isotopes present at lower activity concentrations. LoD of low energy gamma emission affected by high concentrations of higher energy gamma emissions – making peaks more difficult to isolate in spectra

2.3.4 Proportional Counting

Analytical technique	Proportional Counting
Short	-
Description	The proportional counter is a type of gaseous ionization detector device used to measure particles of ionizing radiation.
Emission type	Alpha and Beta
Detector	Gas flow proportional counter
Applicable determinants	Gross alpha and gross beta activities. May also be used for final measurement of nuclides such as Sr-90, Cl-36 following radiochemical separation as an alternative to LSC.
Advantages / Disadvantages	Relatively rapid screening method. Cannot provide information on what isotopes may be present. Poor detection efficiency for low energy betas (e.g., H-3, C-14, Fe-55, Pu-241).
Performance measures (LoD, uncertainties)	LoDs typically in range 0.01 to 0.5 Bq/g.
Pitfalls	Result dependent on calibration nuclides used. Screening technique only - caution required when comparing gross results with sum of individual nuclides in a sample. Some matrices can be analysed directly (e.g., concretes, soils), some may require acid leach or dissolution (e.g., metals).

2.3.5 Inductively Coupled Plasma (ICP)- mass spectrometry (MS)

Analytical technique	Inductively Coupled Plasma (ICP)- mass spectrometry (MS)
Short	ICP-MS
Description	ICP-MS (Inductively Coupled Plasma Mass Spectrometry) is a highly sensitive analytical technique used to measure the elemental composition of a sample. In ICP-MS, a sample is introduced into an inductively coupled plasma (ICP), which is a high-temperature ionized gas, where the sample is ionized and atomized. The resulting ions are separated and measured by their mass-to-charge ratio using a mass spectrometer. ICP-MS is highly sensitive, allowing for the detection of trace amounts of elements, often at levels below parts-per-trillion. It is also highly versatile, able to analyse a wide range of elements across the periodic table, from lithium to uranium.
Emission type	Stable / Chemical
Detector	Mass spectrometer
Applicable determinants	Stable metals / cations. Long half-life radionuclides. (e.g., U-238, U-236, U-235, U-234, U-233, Pu-239, Pu-240, Np-237, Tc-99).
Advantages / Disadvantages	Rapid method – short measurement time. Generally lower LoDs than ICPOES (some exceptions – including K, Ca, Se). Can determine Pu-239 and Pu240 individually. Can determine several of the uranium isotopes individually, including U-233, U- 235 and U236. LoD increases with decreasing half-life. Lower tolerance to dissolved solids than ICP-OES so samples may require dilution. Up to 80 elements can be determined. isobaric interference (the result of equal mass isotopes of different elements present in the sample solution) increases measurement LoD for some radionuclides, e.g., Sr-90, Cs-137
Performance measures (LoD, uncertainties)	LoDs from parts-per-billion down to parts-per-trillion levels.
Pitfalls	-

2.3.6 ICP-Optical Emission Spectrometry (OES)

Analytical technique	ICP-Optical Emission Spectrometry (OES)
Short	ICP-OES
Description	Inductively Coupled Plasma Optical Emission spectroscopy (ICP-OES) is an analytical technique used to determine how much of certain elements are in a sample. The ICP-OES principle uses the fact that atoms and ions can absorb energy to move electrons from the ground state to an excited state.
Emission type	Stable / Chemical
Detector	Charged coupled devices
Applicable determinants	Stable metals / cations
Advantages / Disadvantages	Rapid method – short measurement time. Cannot determine uranium isotopes individually. Higher tolerance to dissolved solids than ICP-MS Simpler and lower cost than ICP-MS. Up to 73 elements measurable.
Performance measures (LoD, uncertainties)	Metals typically 1-10 mg/kg.
Pitfalls	High potential for spectral interference

2.3.7 Gas Chromatography (GC)-MS

Analytical technique	Gas Chromatography (GC)-MS
Short	GC-MS
Description	Gas chromatography–mass spectrometry (GC-MS) is an analytical method that combines the features of gas-chromatography and mass spectrometry to identify different substances within a test sample.
Emission type	Stable / Chemical
Detector	Mass spectrometer
Applicable determinants	Organics (TPH, VOC, SVOC, Polychlorinated Biphenyls (PCB), Polycyclic Aromatic Hydrocarbons (PAH)).
Advantages / Disadvantages	Less sensitive than GC-FID, but preferred where identification of unknowns is required.
Performance measures (LoD, uncertainties)	PCBs 0.5 µg/kg. PAH, SVOC 0.1 mg/kg. VOC 1-5 µg/kg.
Pitfalls	-

2.3.8 GC- Flame Ionisation Detector (FID)

Analytical technique	GC- Flame Ionisation Detector (FID)
Short	GC-FID
Description	Gas Chromatography-Flame Ionization Detection (GC-FID) is an analytical technique that is used to separate and analyze mixtures consisting of volatile components. GC-FID is particularly useful in detecting and quantifying organic compounds.
Emission type	Stable / Chemical
Detector	FID
Applicable determinants	Organics (TPH, VOC).
Advantages / Disadvantages	Improved sensitivity and reproducibility over GC-MS.
Performance measures (LoD, uncertainties)	TPH LoD typically 1 mg/kg.
Pitfalls	-

2.3.9 Ion Chromatography

Analytical technique	Ion Chromatography
Short	IC
Description	Ion Chromatography is a method for separating ions based upon their interactions with resin (stationary phase) and the eluent (mobile phase). These phases differ between an anion column, which attracts anions, and a cation column, which attracts cations.
Emission type	Stable / Chemical
Detector	Conductivity, electrochemical, variable wavelength, photodiode array ion chromatography detector, or mass spectrometer.
Applicable determinants	Anions (e.g. chloride, fluoride, nitrate, nitrite, sulphate). Cations (including alkali metals, alkaline earth metals, heavy metals, transition metals and rare earth metals). Complexants used in decontamination, such as oxalate, EDTA
Advantages / Disadvantages	Can provide information on speciation in addition to quantification. Technique does not suffer from spectral interferences.
Performance measures (LoD, uncertainties)	LoDs in the order of 1 mg/kg.
Pitfalls	-

2.3.10 High- Performance Liquid Chromatography (HPLC)

Analytical technique	High- Performance Liquid Chromatography (HPLC)
Short	HPLC
Description	High-performance liquid chromatography (HPLC) is an analytical technique to separate, identify, and quantify components in a mixture.
Emission type	Stable / Chemical
Detector	Ultra-violet / Visible light, photodiode array. Can be linked (following separation) to ion chromatography detector or mass spectrometry
Applicable determinants	Organics, in particular but not exclusively non-volatile organics. In the context of this GPG, these include hazardous organics, complexants and explosive residues.
Advantages / Disadvantages	Quick and automated, but can be costly and complex for some determinants. Can be complex to identify problems or develop new methods. Some substances irreversibly adsorbed, and hence cannot be detected. If components within the solution are not visible by colouring the mixture or ultraviolet radiation, efficiency of the separation can be difficult to determine.
Performance measures (LoD, uncertainties)	LoDs variable, dependent on detection method. From µg/kg to mg/kg
Pitfalls	Related to complexity and appropriateness of assay techniques, and purity of solvents and reagents

2.3.11 Atomic Absorption Spectrometry

Analytical technique	Atomic Absorption Spectrometry
Short	AAS
Description	Atomic absorption spectrometry (AAS) detects elements in either liquid or solid samples through the application of characteristic wavelengths of electromagnetic radiation from a light source. Individual elements will absorb wavelengths differently, and these absorbances are measured against standards.
Emission type	Stable / Chemical
Detector	-
Applicable determinants	-
Advantages / Disadvantages	#N/A - Collecting information
Performance measures (LoD, uncertainties)	#N/A - Collecting information
Pitfalls	-

2.3.12 Glow Discharge Mass Spectrometry

Analytical technique	Glow Discharge Mass Spectrometry
Short	GDMS
Description	Glow Discharge Mass Spectrometry (GDMS) enables the elemental analysis of solid samples by sputtering in a low-pressure DC argon discharge. The sputtered atoms are ionized in this plasma and extracted into the mass spectrometer for separation and detection.
Emission type	Stable / Chemical
Detector	-
Applicable determinants	-
Advantages / Disadvantages	#N/A - Collecting information
Performance measures (LoD, uncertainties)	#N/A - Collecting information
Pitfalls	-

2.3.13 Time-of-Flight Mass Spectrometry

Analytical technique	Time-of-Flight Mass Spectrometry
Short	TOF-MS
Description	Time-of-Flight (TOF) is a mass analyser that utilises an electric field to accelerate generated ions through the same electrical potential, and then measures the time each ion takes to reach the detector.
Emission type	Stable / Chemical
Detector	0
Applicable determinants	0
Advantages / Disadvantages	#N/A - Collecting information
Performance measures (LoD, uncertainties)	#N/A - Collecting information
Pitfalls	-

2.3.14 Laser Ablation Inductively Coupled Plasma Mass Spectrometry

Analytical technique	Laser Ablation Inductively Coupled Plasma Mass Spectrometry
Short	LA-ICP-MS
Description	LA-ICP-MS (Laser Ablation Inductively Coupled Plasma Mass Spectrometry) is a powerful analytical technology that enables highly sensitive elemental and isotopic analysis to be performed directly on solid samples. LA-ICP-MS begins with a laser beam focused on the sample surface to generate fine particles – a process known as Laser Ablation. The ablated particles are then transported to the secondary excitation source of the ICP-MS instrument for digestion and ionization of the sampled mass. The excited ions in the plasma torch are subsequently introduced to a mass spectrometer detector for both elemental and isotopic analysis.
Emission type	Stable / Chemical
Detector	-
Applicable determinants	-
Advantages / Disadvantages	#N/A - Collecting information
Performance measures (LoD, uncertainties)	#N/A - Collecting information
Pitfalls	-

2.3.15 Resonance Ionization Mass Spectrometry

Analytical technique	Resonance Ionization Mass Spectrometry
Short	RIMS
Description	RIMS (Resonance Ionization Mass Spectrometry) is a type of mass spectrometry that uses resonance ionization to produce ions from a sample. In RIMS, a sample is exposed to a series of laser pulses, causing the selective ionization of specific elements. The ions produced are then separated based on their mass-to-charge ratio in a magnetic sector analyser and detected. The technique is particularly useful for analysing isotopes of elements that are difficult to detect using other techniques, such as radioactive isotopes or isotopes with low natural abundance. RIMS has a wide range of applications, including in nuclear forensics, environmental monitoring, and materials science. The high sensitivity of RIMS allows for the detection of trace amounts of elements, often at levels below parts-per-trillion, making it a valuable tool for a wide range of analytical challenges.
Emission type	Stable / Chemical
Detector	-
Applicable determinants	-
Advantages / Disadvantages	#N/A - Collecting information
Performance measures (LoD, uncertainties)	#N/A - Collecting information
Pitfalls	-

2.3.16 Sector Field Inductively Coupled Plasma Mass Spectrometry

Analytical technique	Sector Field Inductively Coupled Plasma Mass Spectrometry
Short	SF-ICP-MS
Description	SF-ICP-MS (Sector Field Inductively Coupled Plasma Mass Spectrometry) is a type of inductively coupled plasma mass spectrometry (ICP-MS) that uses a magnetic sector as the mass analyser. SF-ICP-MS combines the high sensitivity and accuracy of ICP-MS with the improved mass resolution of a magnetic sector analyser, making it a powerful tool for trace analysis. Compared to conventional ICP-MS, SF-ICP-MS provides improved mass resolution, which can greatly enhance the accuracy and precision of trace analysis. Additionally, it is capable of simultaneously detecting multiple isotopes of the same element, making it ideal for isotope ratio analysis. SF-ICP-MS is more complex and expensive than conventional ICP-MS and requires specialized training and equipment to perform.
Emission type	Stable / Chemical
Detector	-
Applicable determinants	-
Advantages / Disadvantages	#N/A - Collecting information
Performance measures (LoD, uncertainties)	#N/A - Collecting information
Pitfalls	-

2.3.17 Thermal Ionization Mass Spectrometry

Analytical technique	Thermal Ionization Mass Spectrometry
Short	TIMS
Description	TIMS (Thermal Ionization Mass Spectrometry) is a type of mass spectrometry that uses thermal ionization to produce ions from a sample. In TIMS, a sample is heated to a high temperature, causing the release of ions that are then separated based on their mass-to-charge ratio in a magnetic sector analyser. The ions are then detected and their quantity is determined, providing information about the isotopes present in the sample. TIMS is widely used for the analysis of trace elements and isotopes in geological materials and for the determination of isotopic ratios. TIMS is particularly useful for measuring isotopic ratios of elements with high atomic masses, such as uranium, thorium, and lead, and for analysing very small samples, often in the sub-microgram range. TIMS is widely used in geochronology and isotope geochemistry, as well as in nuclear forensics and environmental science, where it can provide valuable information about the sources and fate of pollutants in the environment.
Emission type	Stable / Chemical
Detector	-
Applicable determinants	-
Advantages / Disadvantages	#N/A - Collecting information
Performance measures (LoD, uncertainties)	#N/A - Collecting information
Pitfalls	-

2.3.18 Accelerator Mass Spectrometry

Analytical technique	Accelerator Mass Spectrometry
Short	AMS
Description	AMS (Accelerator Mass Spectrometry) is a type of mass spectrometry that uses an accelerator to increase the energy of ions prior to analysis. This allows for the detection of very low levels of isotopes, making it an extremely sensitive method. In AMS, a sample is first ionized and the ions are then accelerated to high energies and passed through a magnetic field to separate them based on their mass-to-charge ratio. The ions are then detected and their quantity is determined, providing information about the isotopes present in the sample. AMS is widely used for the analysis of radioisotopes, including carbon-14, and for the determination of trace levels of elements in environmental and biological samples.
Emission type	Stable / Chemical
Detector	-
Applicable determinants	-
Advantages / Disadvantages	Complex system with ion-source, accelerator and detector array. Separation and purification of isotope to be measured is usually needed.
Performance measures (LoD, uncertainties)	-

2.4 Physical parameters

Property	Measurement method
Density	The density at any point of a homogeneous waste equals its total mass divided by its total volume. The mass is normally measured with an appropriate scale or balance; the volume may be measured directly (from the geometry of the object) or by the displacement of a fluid. For determining the density of a liquid, a hydrometer may be used. If the waste is not homogeneous, then the density is a function of the position. In that case the density around any given location is determined by calculating the density of a small volume around that location. The density of granular waste can be ambiguous, depending on exactly how its volume is defined, and this may cause confusion in measurement. A common example is sand: if it is gently poured into a container, the density will be low; if the same sand is then compacted, it will occupy less volume and consequently exhibit a greater density. This is because sand, like all powders and granular solids, contains a lot of air space in between individual grains. The density of the material including the air spaces is the bulk density, which differs significantly from the density of an individual grain of sand with no air included.
Free liquids	Free liquids can potentially migrate through the environment and have the potential to leach out dissolved radioactive substances to the surrounding environment. It increases the rate of corrosion, the waste package degrades faster and also increases the rate of gas formation, which can also mobilize radionuclides and in some cases may threaten the mechanical stability of the waste mass and even the entire storage. In this framework, assessment of the process giving rise to the waste (i.e. process knowledge) is essential to identify the steps of the process where free liquid might be generated with waste. Then, during waste generation, the correct implementation of the quality assurance system by the waste generator should avoid presence of free liquids in raw waste. Presence of free liquid in raw waste may be also be physically assessed by: • Visually, e.g. when retrieving historical waste from trenches, presence of rust; bags with raw waste with visible moisture or free liquids, etc. • by digital radiography, e.g. if raw waste is pre-packed in drums before compaction;
Moisture content	The classic laboratory method of measuring high level moisture in solid or semi-solid waste is loss on drying (LOD). In this technique a sample of waste is weighed, heated in an oven for an appropriate period, cooled in the dry atmosphere of a desiccator, and then reweighed. If the volatile content of the solid is primarily water, the LOD technique gives a good measure of moisture content. Because the manual laboratory method is relatively slow, automated moisture analyzers have been developed that can reduce the time necessary for a test from a couple hours to just a few minutes. These analysers incorporate an electronic

	balance with a sample tray and surrounding heating element. Under microprocessor control the sample can be heated rapidly and a result computed prior to the completion of the process, based on the moisture loss rate, known as a drying curve. The exudation of water from waste form sample under compression. This test technique describes a method to evaluate the exudation of water under compressive stress on samples of homogeneous waste form contained in package of radioactive waste. The test applies to blocks of homogeneous waste form with a polymer matrix or bitumen as immobilization material. It is based on the application to the block of waste (without its container) placed in vertical position, of a load of 0.35 MPa and evaluation of the liquid exuded and the variation of the mass of the block. In case of waste packages these tests can be performed on samples of the external container without giving any credit to the waste form performance.
Porosity	Test used to measure this criterion is to percolate a gas under a predetermined pressure through a cylindrical specimen of concrete or mortar, to measure the flow and to deduce the permeability of the material tested. The technique allows measurement of permeability greater than 10-19m2. The water content of the material having an important influence on the transfer of gas permeability measurement is performed at different saturation levels. The tests are performed on representative (real or simulated) samples of components based hydraulic binder involved. The real samples can be obtained either on the hydraulic binder production line (moulded) or cores made on real waste package. For all types of hydraulic binders and regardless of the type of cement used, the test must be performed on samples with a long time of curing (e.g. 90 days or more).

2.5 Chemical parameters

Property	Measurement method
Chelating and complexion agents	The most pertinent chemical properties, with a direct impact on the repository (surface disposal), to determine in solid waste are: water-soluble phosphates & chlorides, fluorine, sulphate, electropositive metals, cellulose content and pH (of absorbed fluids). For any waste containing concrete rubble, the Alkali-Silica Reaction (ASR) must be excluded. ASR must also be excluded for any material used in a cement-based immobilization matrix for conditioned waste (test method: ASTM C 1260 or 1293). Similarly, also Delayed Ettringite Formation (DEF) needs to be excluded in concrete rubble or in the immobilization matrix. Sensitivity to DEF in concrete rubble can e.g., be detected using petrography. Both reactions create a gel that might put pressure causing the cementitious conditioning matrix to crack. Other properties (e.g., dimensions, type of polymers) are linked to treatment & conditioning processes. Critical chemical elements are determined in samples, by using ion chromatography which allows the quantification of specific cations or anions. At SCK CEN, three instruments are available each with specific possibilities. F-, Cl-, Br-, I-, NO3-, SO32-, SO4 2-, S2O32-, NH4+, saccharides, oxalate, acetate and formate (HCO2-) can be quantified. This technique is mainly used for characterization of reactor water, and for samples coming from effluents and packed liquid waste. To a lesser extent, it is also applied to samples coming from solid waste. For the treatment at Belgoprocess of operational effluents, another important parameter that needs to be determined is COD (Chemical oxygen demand). The technique applied is the COD Cell Test (Spectroquant). Here, the water sample is oxidized with a hot sulfuric solution of potassium dichromate, with silver sulphate as the catalyst. Chloride is masked with mercury sulphate. The consumption of dichromate during the oxidation process is photometrically determined, by measuring the concentration of green Cr3+ ions at a wavelength of 600 nm.
Corrosive materials	The most pertinent chemical properties, with a direct impact on the repository (surface disposal), to determine in solid waste are: water-soluble phosphates & chlorides, fluorine, sulphate, electropositive metals, cellulose content and pH (of absorbed fluids). For any waste containing concrete rubble, the Alkali-Silica Reaction (ASR) must be excluded. ASR must also be excluded for any material used in a cement-based immobilization matrix for conditioned waste (test method: ASTM C 1260 or 1293). Similarly, also Delayed Ettringite Formation (DEF) needs to be excluded in concrete rubble or in the immobilization matrix. Sensitivity to DEF in concrete rubble can e.g., be detected using petrography. Both reactions create a gel that might put pressure causing the cementitious conditioning matrix to crack. Other properties (e.g., dimensions, type of polymers) are linked to treatment & conditioning processes. Critical chemical elements are determined in samples, by using ion chromatography which allows the quantification of specific cations or anions. At SCK CEN, three instruments are available each with specific possibilities. F-, Cl-, Br-, I-, NO3-, SO32-, SO4 2-, S2O32-, NH4+, saccharides, oxalate, acetate and formate (HCO2-) can be quantified. This technique is mainly used for characterization of reactor water, and for samples coming from effluents and packed liquid waste. To a lesser extent, it is also applied to samples coming from solid waste. For the treatment at Belgoprocess of operational effluents, another important

	parameter that needs to be determined is COD (Chemical oxygen demand). The technique applied is the COD Cell Test (Spectroquant). Here, the water sample is oxidized with a hot sulfuric solution of potassium dichromate, with silver sulphate as the catalyst. Chloride is masked with mercury sulphate. The consumption of dichromate during the oxidation process is photometrically determined, by measuring the concentration of green Cr3+ ions at a wavelength of 600 nm.
Hazardous materials	The most pertinent chemical properties, with a direct impact on the repository (surface disposal), to determine in solid waste are: water-soluble phosphates & chlorides, fluorine, sulphate, electropositive metals, cellulose content and pH (of absorbed fluids). For any waste containing concrete rubble, the Alkali-Silica Reaction (ASR) must be excluded. ASR must also be excluded for any material used in a cement-based immobilization matrix for conditioned waste (test method: ASTM C 1260 or 1293). Similarly, also Delayed Ettringite Formation (DEF) needs to be excluded in concrete rubble or in the immobilization matrix. Sensitivity to DEF in concrete rubble can e.g., be detected using petrography. Both reactions create a gel that might put pressure causing the cementitious conditioning matrix to crack. Other properties (e.g., dimensions, type of polymers) are linked to treatment & conditioning processes. Critical chemical elements are determined in samples, by using ion chromatography which allows the quantification of specific cations or anions. At SCK CEN, three instruments are available each with specific possibilities. F-, Cl-, Br-, I-, NO3-, SO32-, SO4 2-, S2O32-, NH4+, saccharides, oxalate, acetate and formate (HCO2-) can be quantified. This technique is mainly used for characterization of reactor water, and for samples coming from effluents and packed liquid waste. To a lesser extent, it is also applied to samples coming from solid waste. For the treatment at Belgoprocess of operational effluents, another important parameter that needs to be determined is COD (Chemical oxygen demand). The technique applied is the COD Cell Test (Spectroquant). Here, the water sample is oxidized with a hot sulfuric solution of potassium dichromate, with silver sulphate as the catalyst. Chloride is masked with mercury sulphate. The consumption of dichromate during the oxidation process is photometrically determined, by measuring the concentration of green Cr3+ ions at a wavelength of 600 nm.

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