Strategy and methodology for NORM characterization

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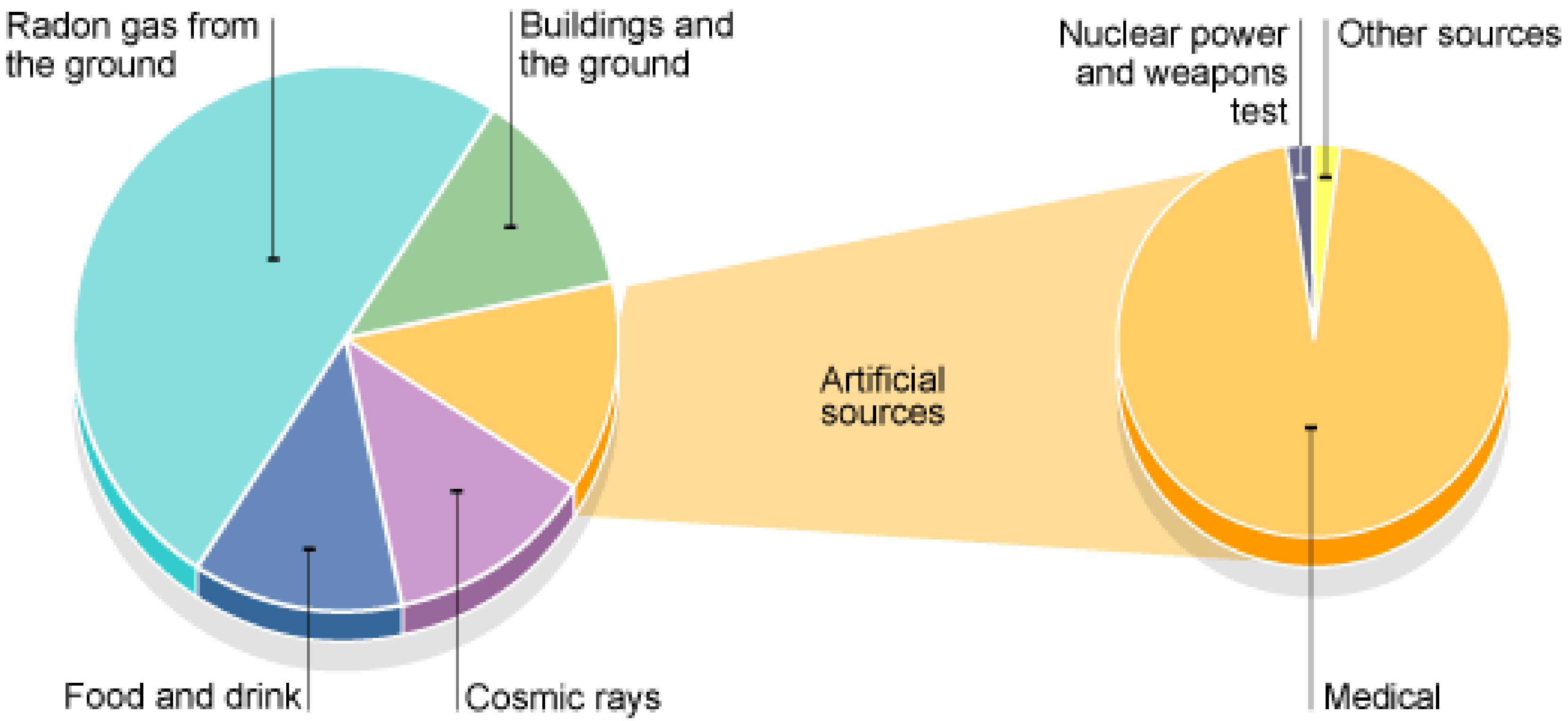
Origins of NORM

- Radionuclides of natural origin are present in most material, the most common being the radionuclides of the **^{238}U and ^{232}Th series and ^{40}K**.

- This material is commonly referred to as **Naturally Occurring Radioactive Material (NORM).**

- In some materials **activity concentrations** of radionuclides of natural origin are **significantly elevated**, to the extent that **regulatory control may be required** for radiation protection purposes.

Average annual dose UK



Source:
http://www.bbc.co.uk/schools/gcsebitesize/science/add_aqa/atoms_radiation/nuclearradiationrev1.shtml

Comparison of doses from sources of exposure

Source of exposure	Dose
Dental x-ray	0.005 mSv
100g of Brazil nuts	0.01 mSv
Chest x-ray	0.014 mSv
Transatlantic flight	0.08 mSv
Nuclear power station worker average annual occupational exposure (2010)	0.18 mSv
UK annual average radon dose	1.3 mSv
CT scan of the head	1.4 mSv
UK average annual radiation dose	2.7 mSv
USA average annual radiation dose	6.2 mSv
CT scan of the chest	6.6 mSv
Average annual radon dose to people in Cornwall	7.8 mSv
CT scan of the whole spine	10 mSv
Annual exposure limit for nuclear industry employees	20 mSv

https://www.gov.uk/government/publications/ionising-radiation-dose-comparisons/ionising-radiation-dose-comparisons

Origins of NORM

- Production and use of **thorium** and its compounds
- **Mining** of ores
- Production of **oil and gas**
- The **zircon** and zirconia industries
- Manufacture of **TiO_2** pigment
- Production of **phosphate, clay** and building materials
- Production of iron and steel, tin, copper, aluminium, zinc and lead
- **Combustion of fossil fuels-** coal, oil, gas
- **Water treatment**

Origins of NORM

Scale deposited in an oil and gas production tubular

Source: IAEA Tecdoc 1712

NORM radionuclides

Nuclide	decay mode	half life
C-14	β-	5700 y
H-3 (tritium)	β-	12.32 y
Na-22	β+ and electron capture	2.6 y
Be-7	Electron capture	53.22 d

Cosmogenic NORM radionuclides

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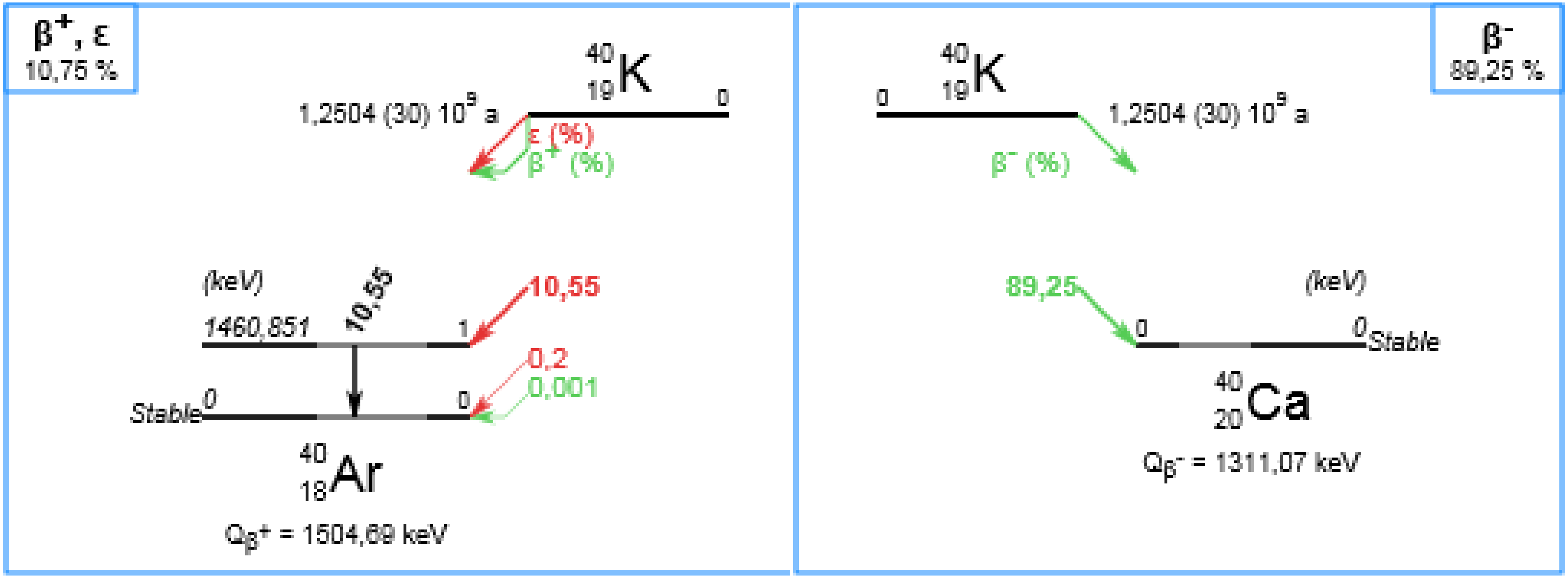
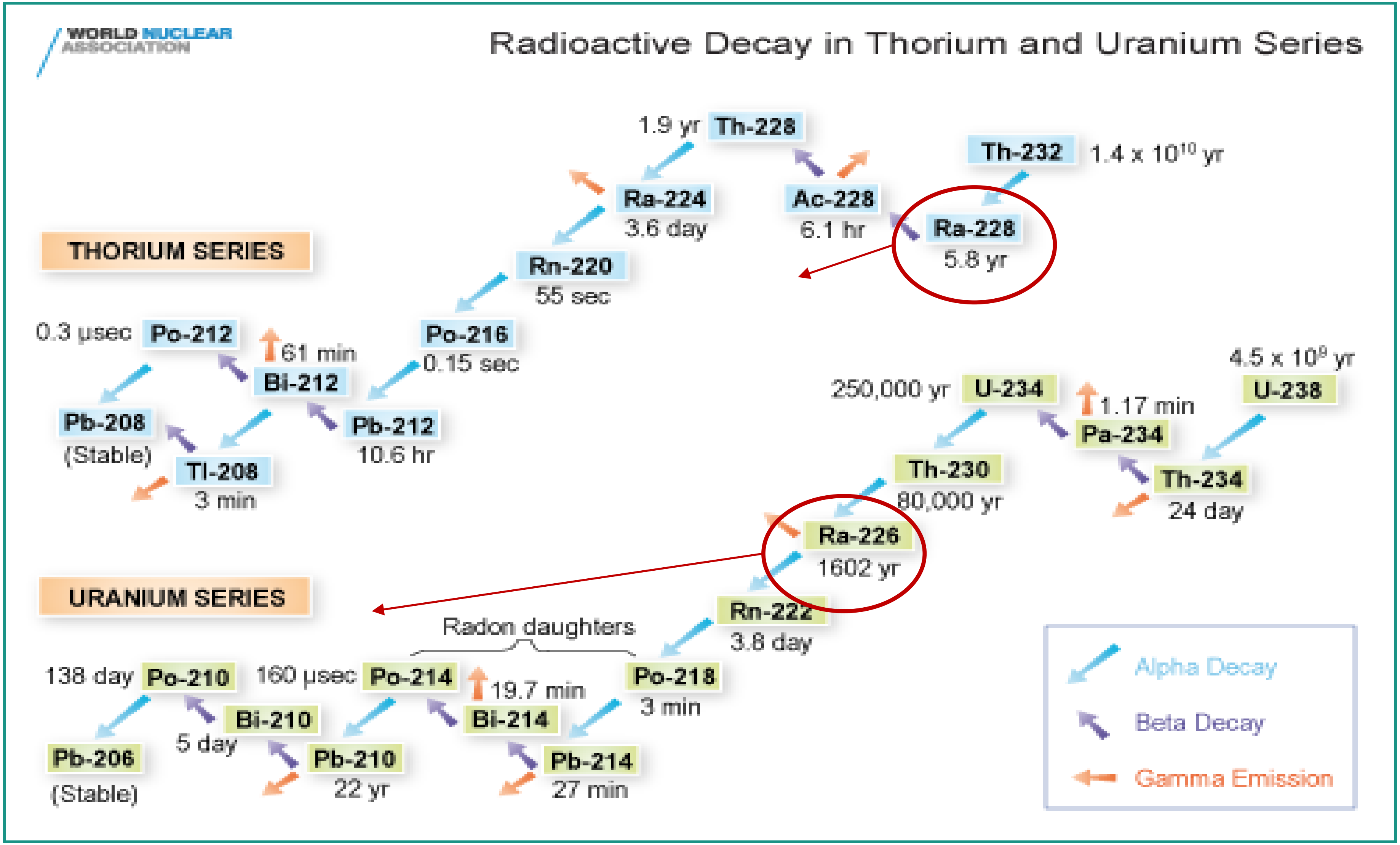
Potassium-40

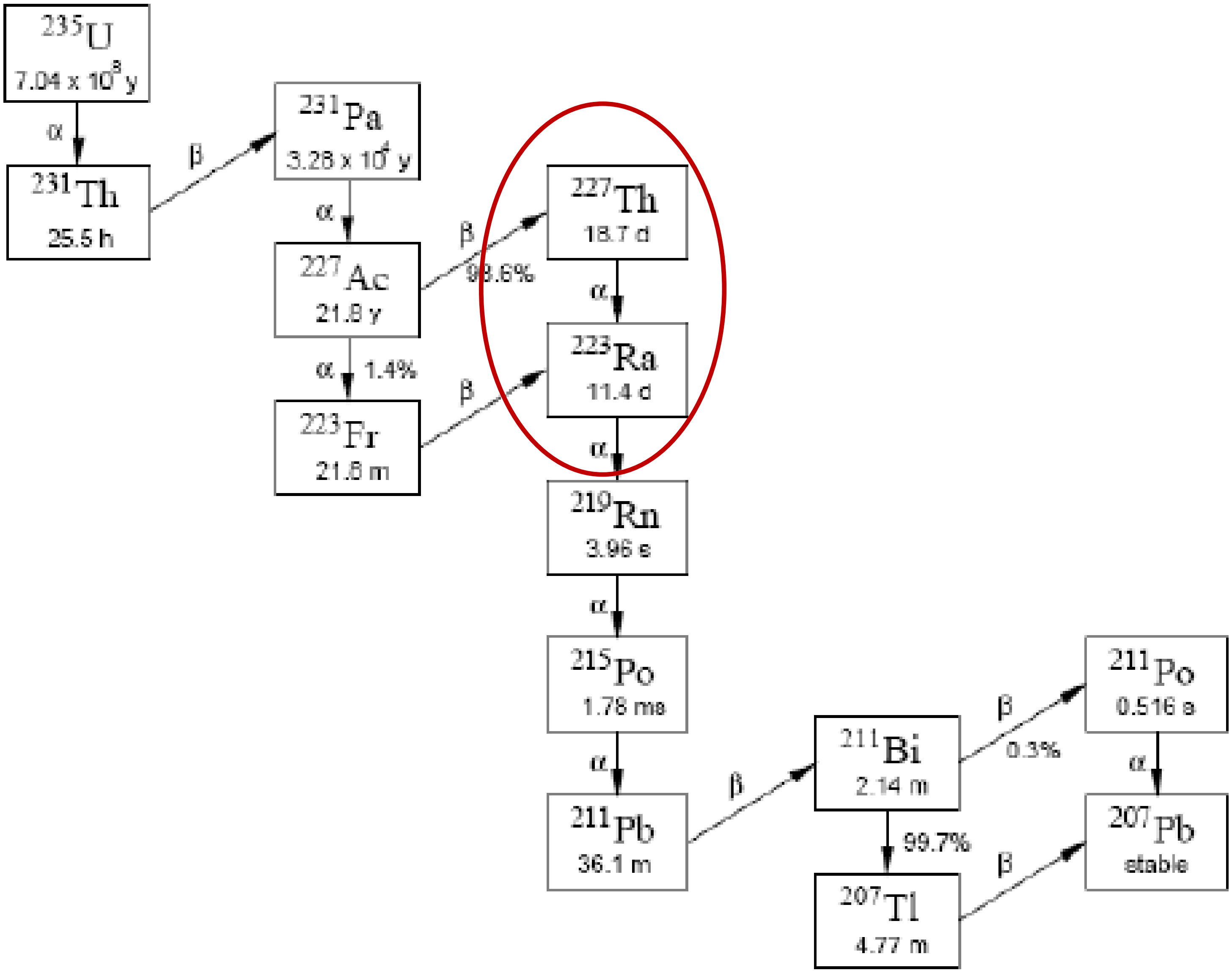


^{40}K decay scheme

NORM radionuclides



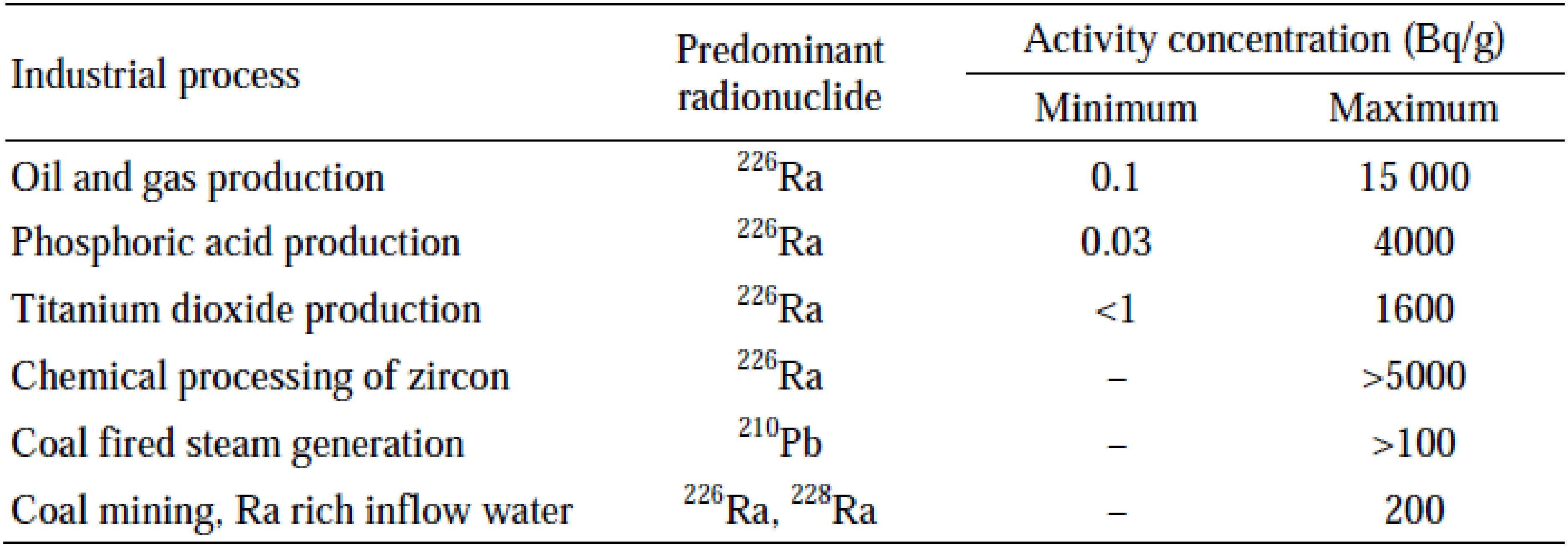
Members of the ^{238}U and ^{232}Th decay chains



Members of the ^{235}U decay chain

Origins of NORM

Typical RN activity concentrations in scale deposits



Industrial process	Predominant radionuclide	Activity concentration (Bq/g)	
		Minimum	Maximum
Oil and gas production	^{226}Ra	0.1	15 000
Phosphoric acid production	^{226}Ra	0.03	4000
Titanium dioxide production	^{226}Ra	<1	1600
Chemical processing of zircon	^{226}Ra	–	>5000
Coal fired steam generation	^{210}Pb	–	>100
Coal mining, Ra rich inflow water	^{226}Ra, ^{228}Ra	–	200

Source: IAEA 2003, Safety Report Series 34.

Origins of NORM

NORM in oil and gas production

Radionuclide	Natural gas Bq/m^3	Produced water Bq/L	Hard scale Bq/kg	Sludge Bq/kg
U-238		trace	1 – 500	5 - 10
Ra-226		0.002 – 1,200	100 – 15 million	50 - 800,000
Po-210	0.002 – 0.08		20 - 1500	4 - 160,000
Pb-210	0.005 – 0.02	0.05 - 190	20 - 75,000	10 - 1.3 million
Rn-222	5 – 200,000			
Th-232		trace	1 - 2	2 - 10
Ra-228		0.3 - 180	50 - 2.8 million	500 - 50,000
Ra-224		0.05 - 40		

Source: IAEA 2003, Safety Report Series 34.

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GUIDANCE ON THE IMPLEMENTATION OF THE WATER SUPPLY (WATER QUALITY) REGULATIONS 2000 (as amended) IN ENGLAND

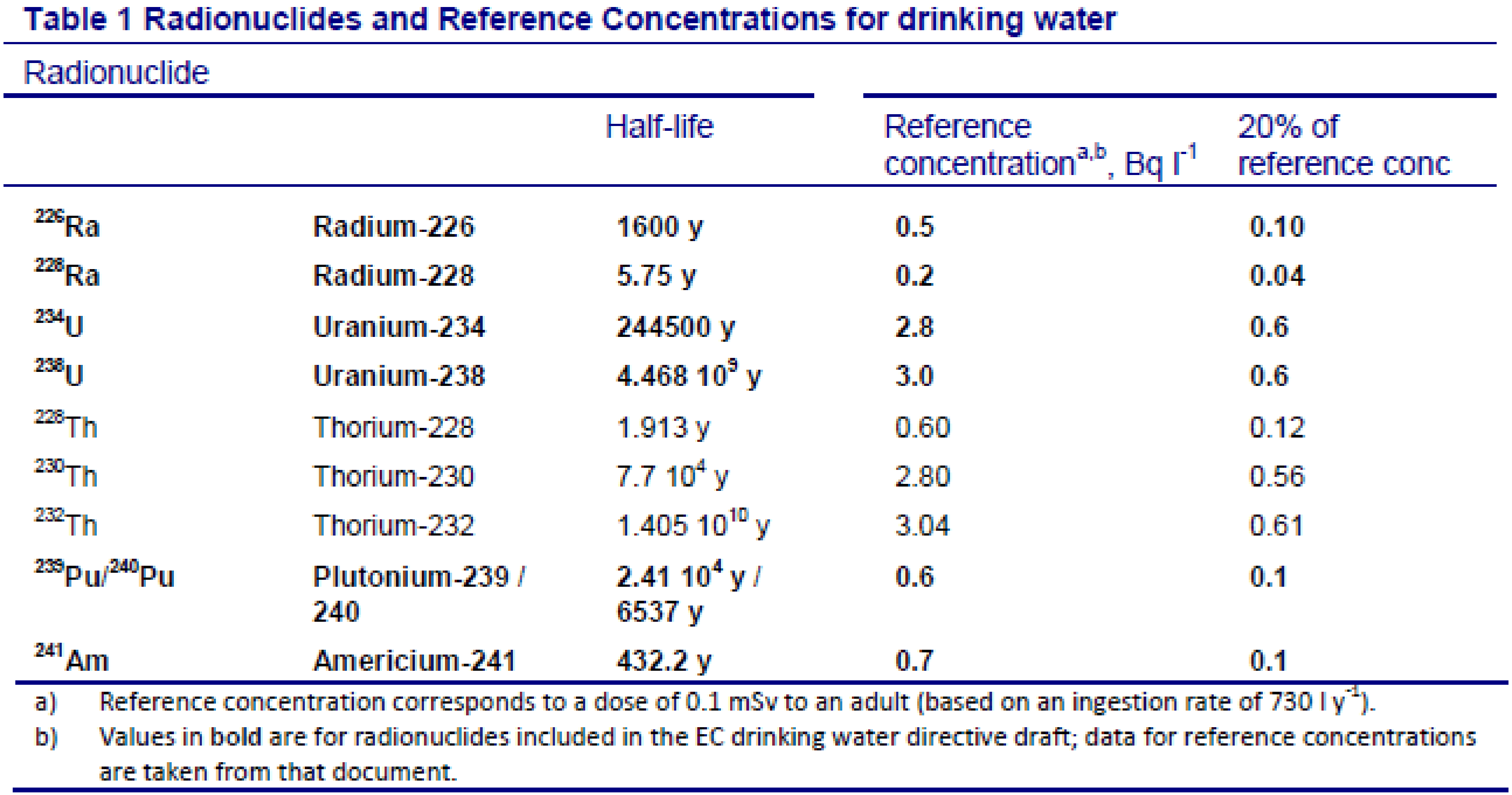
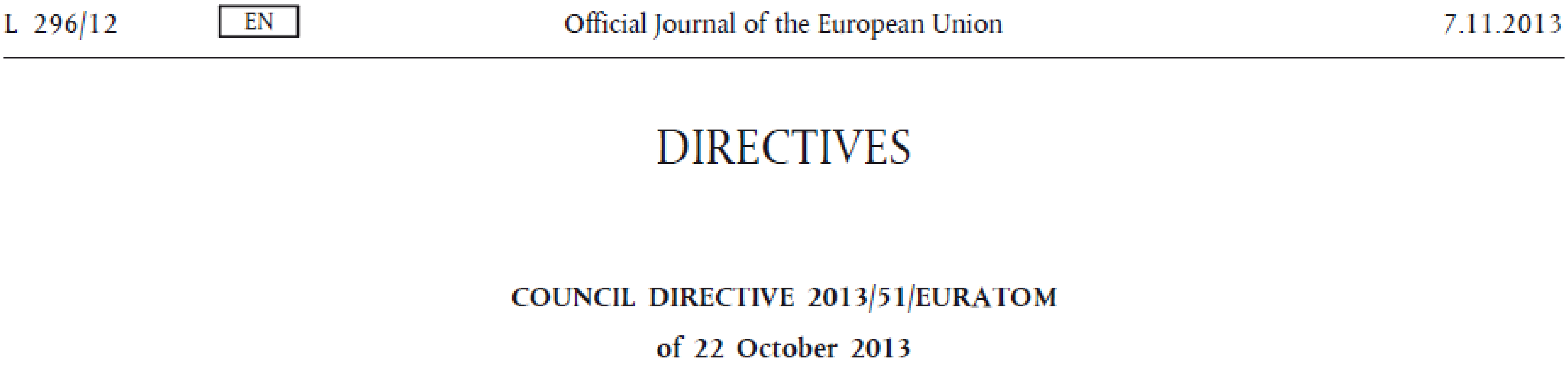


Table 1 Radionuclides and Reference Concentrations for drinking water

Radionuclide		Half-life	Reference concentration[a,b], Bq l^{-1}	20% of reference conc
^{226}Ra	**Radium-226**	**1600 y**	**0.5**	**0.10**
^{228}Ra	**Radium-228**	**5.75 y**	**0.2**	**0.04**
^{234}U	**Uranium-234**	**244500 y**	**2.8**	**0.6**
^{238}U	**Uranium-238**	**4.468 10^9 y**	**3.0**	**0.6**
^{228}Th	Thorium-228	1.913 y	0.60	0.12
^{230}Th	Thorium-230	7.7 10^4 y	2.80	0.56
^{232}Th	Thorium-232	1.405 10^{10} y	3.04	0.61
^{239}Pu/^{240}Pu	**Plutonium-239 / 240**	**2.41 10^4 y / 6537 y**	**0.6**	**0.1**
^{241}Am	**Americium-241**	**432.2 y**	**0.7**	**0.1**

a) Reference concentration corresponds to a dose of 0.1 mSv to an adult (based on an ingestion rate of 730 l y^{-1}).

b) Values in bold are for radionuclides included in the EC drinking water directive draft; data for reference concentrations are taken from that document.



L 296/12 EN Official Journal of the European Union 7.11.2013

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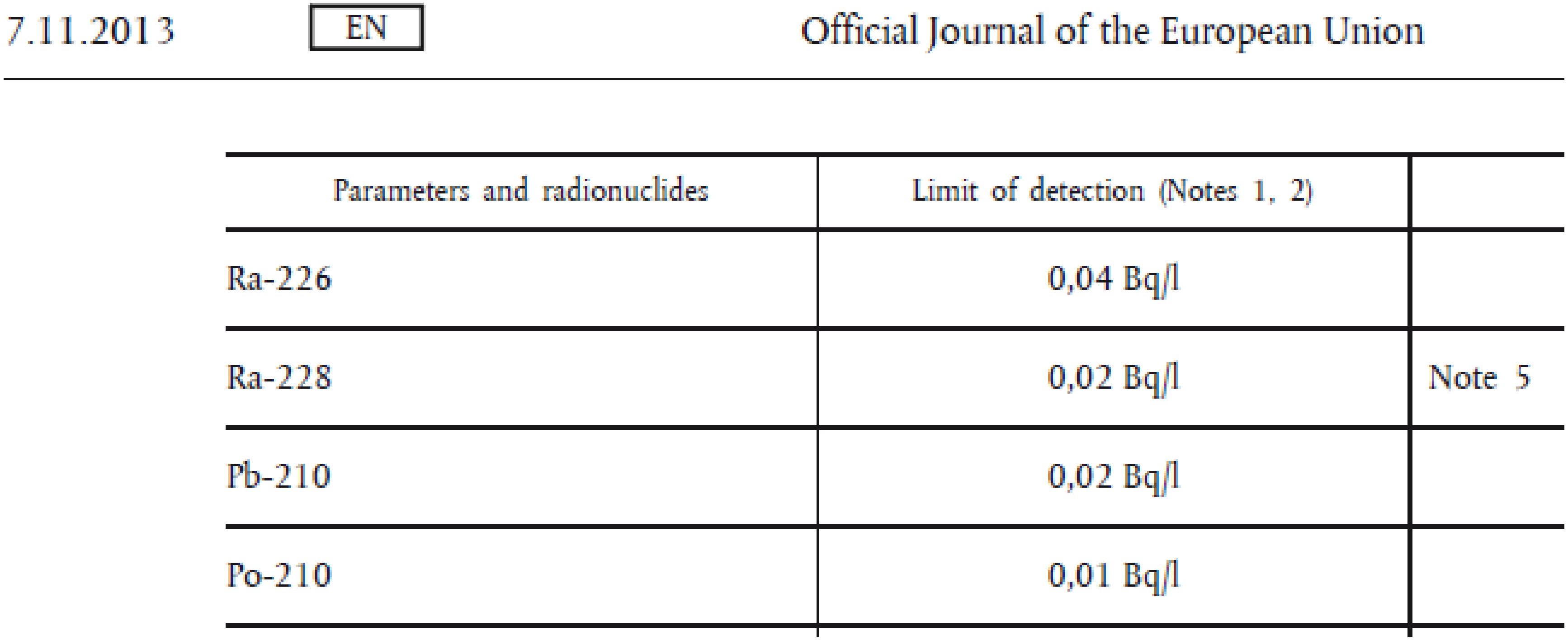
of 22 October 2013



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Parameters and radionuclides	Limit of detection (Notes 1, 2)	
Ra-226	0,04 Bq/l	
Ra-228	0,02 Bq/l	Note 5
Pb-210	0,02 Bq/l	
Po-210	0,01 Bq/l	

Detection limits of Ra-226, Ra-228, Pb-210 and Po-210

Chemistry considerations

- **Th**- oxidation state IV, **low solubility**
- **U**- oxidation state VI, uranyl ion **UO_2^{2-}**, **high solubility**
- Ra- Oxidation state II, high solubility and mobility
- Presence of **SO_4^{2-}** - high U(VI) solubility, low Ra(II) solubility, **$RaSO_4$ precipitates**
- Presence of Cl^- low Pb(II) solubility (**$PbCl_2 \downarrow$**)
- Rn, Po, Pb- high **volatility**
- **Disequilibrium**

Characterisation

Waste **characterisation** strategy is **part of the overall** waste management **strategy**

Waste characterisation strategy is **developed based on**:

- **Waste streams parameters**
- **Waste Acceptance Criteria (WAC)**
- **Process control**
- **Quality assurance** requirements
- **Transportation** requirements
- Workers **safety requirements**

Characterisation objectives

- Establish a **process for characterising** radioactive waste during **all stages**
- Contribute to **safety during different stages:** generation, processing, transport, storage and disposal
- Demonstrate **compliance with WAC**

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Strategy in defining the characterisation programme

The type and extent of the characterization efforts **depend on many factors***:*

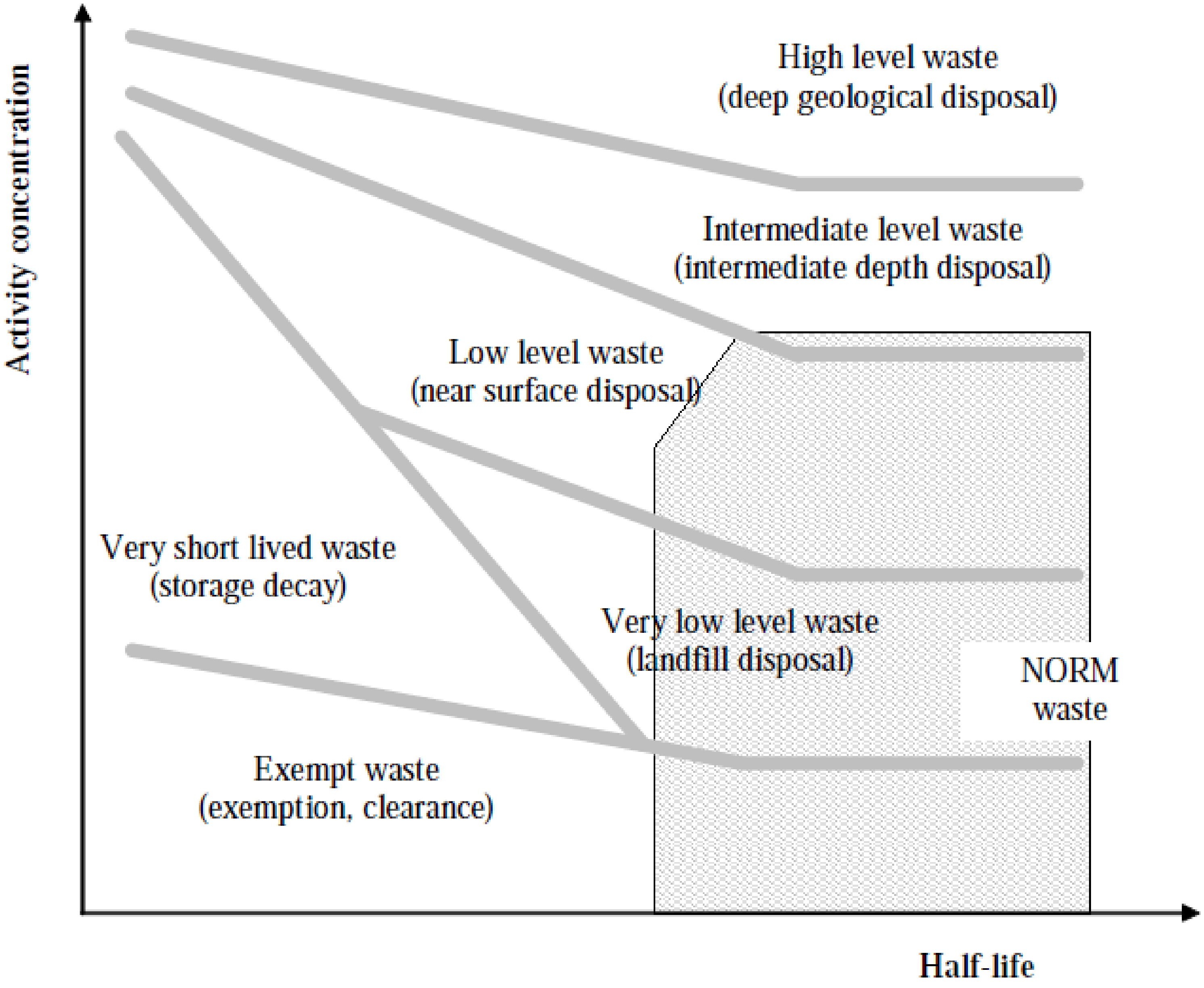
- the **type of waste** or waste form;
- the **disposal concept**;
- the **regulatory regime**;
- the amount of **process knowledge** that is available;
- the **kind of characteristics** to be measured.

Strategy in defining the characterisation programme

It is **not possible** to define or recommend a **single characterization procedure** for even similar waste forms or packages.

- the **waste acceptance criteria** (and the rationale behind);
- the **safety assessment** for the disposal concept (e.g. assumptions made, information needed).

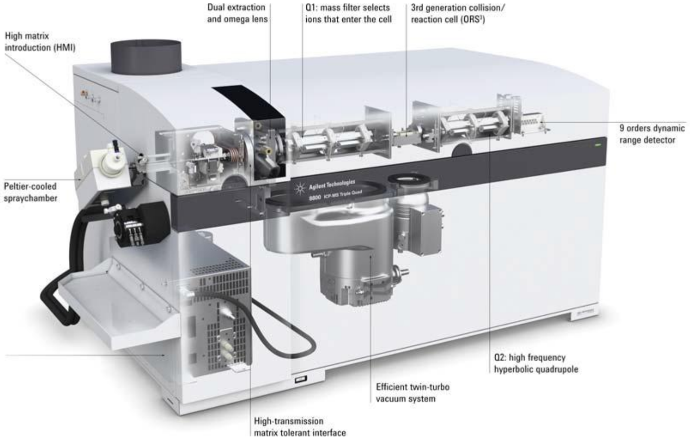
It is of importance that **waste characterization is considered as part of the larger waste management strategy** and interactions should be occurring among the various working groups involved.

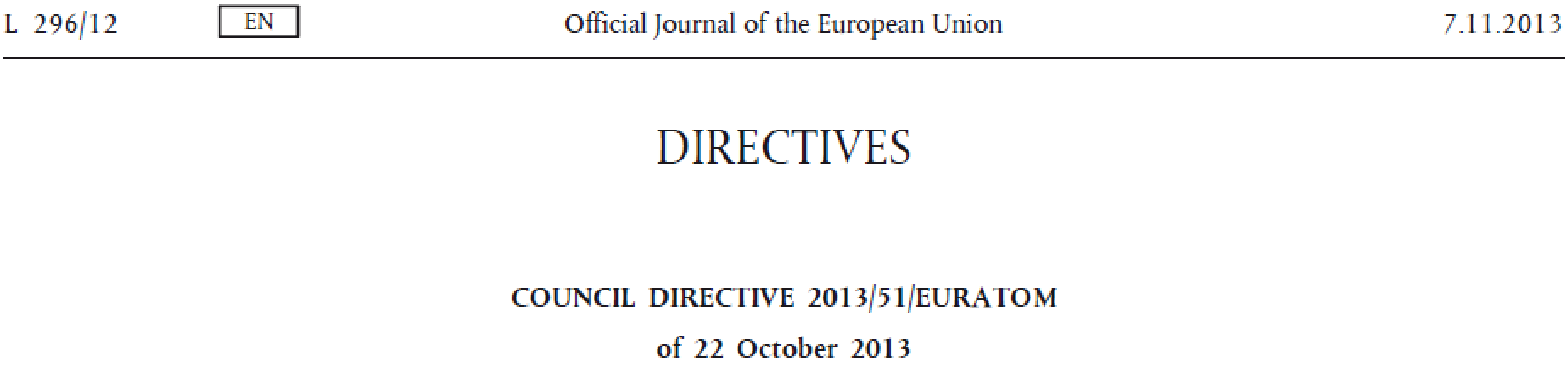


Conceptual illustration of the waste classification scheme

Analysis

- ETM vs. DTM nuclides
- Stages of radionuclide analysis and quantification
- When sample pre-treatment is required
- Types of radiochemistry procedures
- Radiometric counting methods
- Specialised analytical methods





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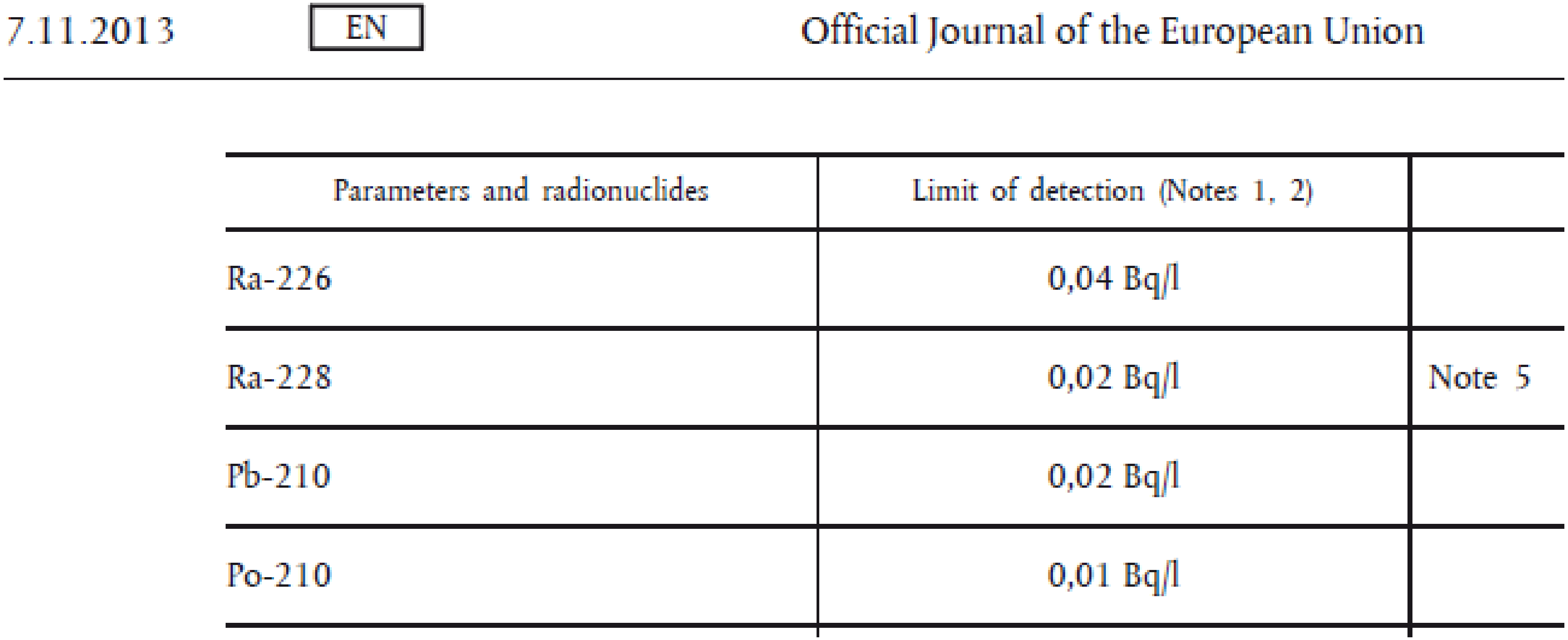

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Po-210	0,01 Bq/l	

Detection limits of Ra-226, Ra-228, Pb-210 and Po-210

Sample preparation

- **Receiving**
- Sample **logging** and tracking
- **Crashing**, drying, homogenising
- Dispensing, **weighting**
- Sample **dissolution** (for destructive analysis)
- **Separation**, preparing of counting **sources** (for NDA)

Tracers and carriers:

Carriers:

- Why ne need to add carriers?

1Bq of Ra-226 ($T_{1/2}$=1600y) is $1.2.10^{-13}$M
1Bq of Zr-95 ($T_{1/2}$=64d) is $1.4.10^{-18}$M

Carriers **are chemically identical** materials to the radionuclide of interest that have a significant, non-radioactive mass.

Losses of radionuclides during the analytical separation processes is due to irreversible adherence to dust, container walls, ion exchange resins, and filters, etc.

If we add a material that is chemically identical to the radionuclide of interest, it will also occupy these sites and **reduce the radionuclide losses**.

The surface becomes saturated mainly with stable strontium atoms because of the vast mass excess above the radioactive atoms.

Carriers

Carriers:

- Isotopic
- Non-Isotopic.

Isotopic carrier is a stable isotope of the radionuclide of interest. For ^{90}Sr, **stable strontium is added** to the sample at the beginning of the sample analysis. Radioactive and stable strontium are chemically identical. If the stable strontium precipitates as the carbonate, the radioactive strontium will precipitate as well because the total mass of strontium present will exceed the solubility product constant for strontium carbonate.

Non-isotopic carrier- different element with similar chemical behaviour, i.e. barium is used in radium analysis.

Tracers

What do we expect from a **tracer**?

The tracer must exhibit the **same chemical behaviour** as the analyte:

-Implies that the same element should be employed

The tracer should **not interfere with the measurement** of the analyte:

-Preferable to measure by the same technique, or if the tracer does not register in the analyte measurement (and *vice versa*)

Chemical equilibrium between the tracer and analyte should be **established at the earliest possible point** in the analysis:

-Add the tracer as soon as possible and (for solids) employ total dissolution

Tracers

Furthermore:

The tracer **should not be present in the samples** being analysed:

-Using nuclides present in the samples being analysed complicates analysis

The tracer should be **pure and not introduce contamination** into samples being measured:

-Purity requirement: may differ for mass spectrometry and radiometric measurements

The tracer **activity should be traceable** to national or international standards:

-Not strictly so: Measurements of γ emitting tracers may be relative

Tracers

U-232 ($T_{1/2}$=68.9y, α)

U-236 ($T_{1/2}$=2.3.10^7y, α)

U-237 ($T_{1/2}$=6.75d, γ, β^-)

Th-227 ($T_{1/2}$=18.7d, α)

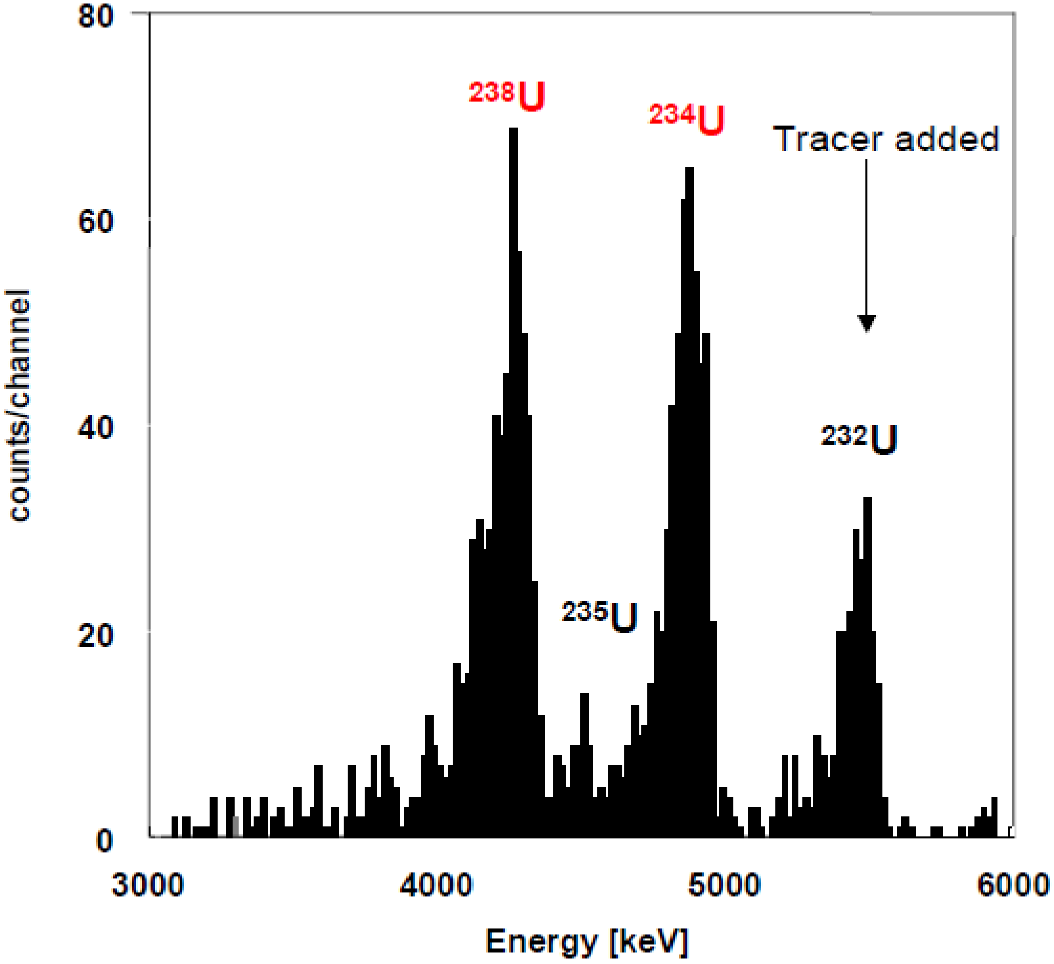
Th-229 ($T_{1/2}$=7340y, α)

Ra225 ($T_{1/2}$=14.9d, β^-)

Ra223 ($T_{1/2}$=11.4d, α)

Po-208 ($T_{1/2}$=2.9y, α)

Po-209 ($T_{1/2}$=102y, α)

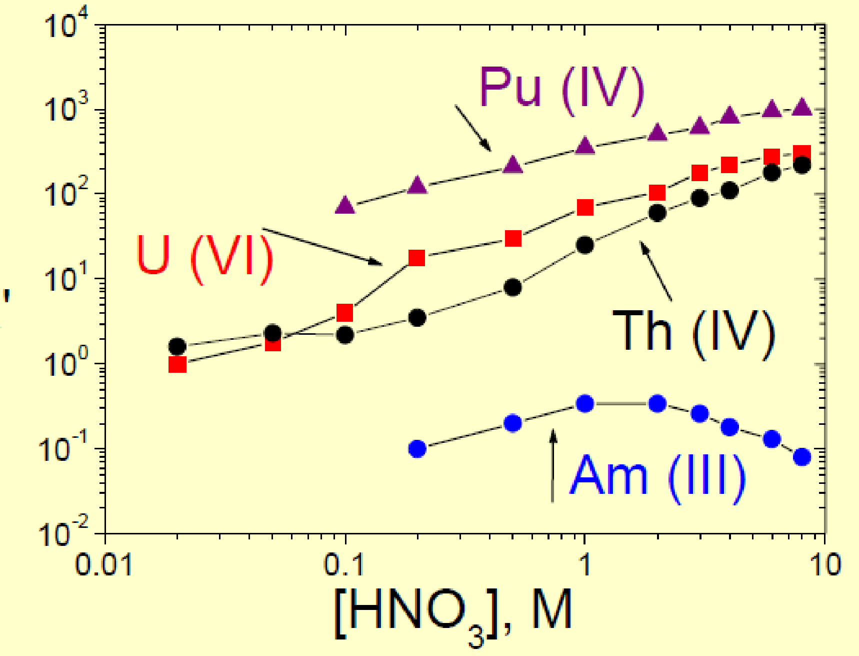


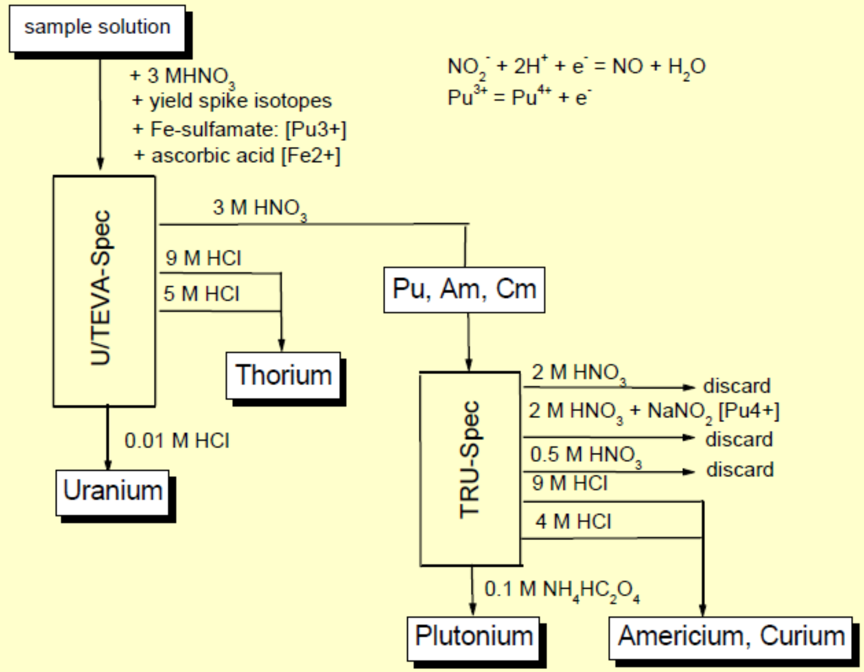
α- Spectrum of ^{nat}U with ^{232}U tracer added

Radiochemical pre-treatment

Radiochemical separations:

- Precipitation
- Solvent extraction;
- Ion-exchange chromatography;
- Extraction chromatography





sample solution

+ 3 M HNO_3
+ yield spike isotopes
+ Fe-sulfamate: [Pu3+]
+ ascorbic acid [Fe2+]

$NO_2^- + 2H^+ + e^- = NO + H_2O$
$Pu^{3+} = Pu^{4+} + e^-$

U/TEVA-Spec

3 M HNO_3 → Pu, Am, Cm

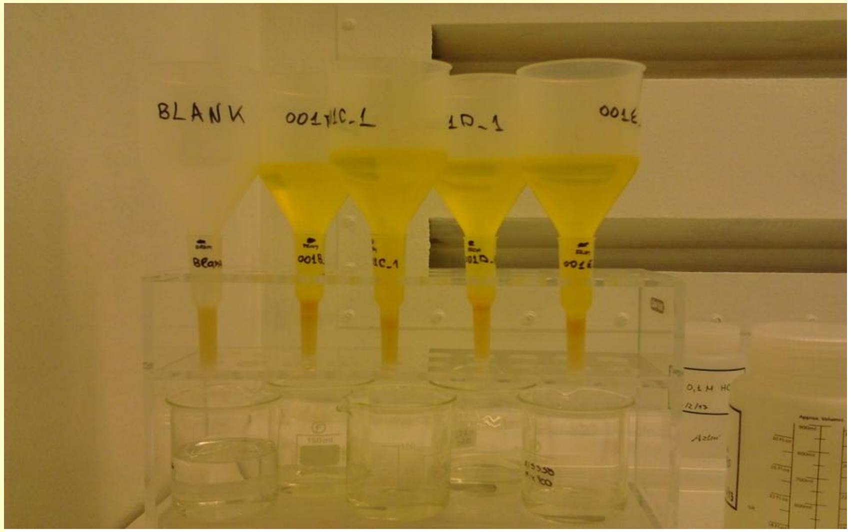
9 M HCl
5 M HCl → Thorium

0.01 M HCl → Uranium

TRU-Spec

2 M HNO_3 → discard
2 M HNO_3 + $NaNO_2$ [Pu4+] → discard
0.5 M HNO_3 → discard
9 M HCl
4 M HCl → Americium, Curium

0.1 M $NH_4HC_2O_4$ → Plutonium



Precipitation

$CaCO_3$

Pre-concentration of **Sr from aqueous samples**

Ca-oxalate

Sr from large seawater samples

Ca-phosphate

Oxalate precipitation is frequently used in the **pre-concentration of actinides and Sr^{2+}** (and Y^{3+}), and to remove interfering elements as K^+ and Fe^{3+} as they are left in the solution.

$BaSO_4$

Pre-concentration and **separation of Ra**

LaF_3

Co-precipitate with **actinides in oxidation state III and IV**, and do not co-precipitate actinides in oxidation state V and VI.

$Fe(OH)_3$

Co-precipitation of actinides is extensively used to pre-concentrate **actinides from large aqueous sample volumes**

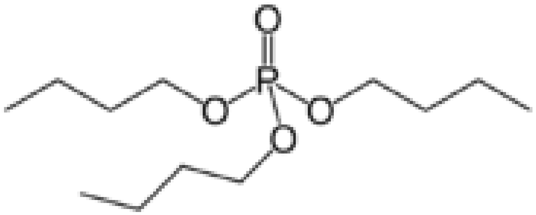
MnO_2

Scavenges **actinides in III and IV oxidation state as well as Ra**

Solvent extraction

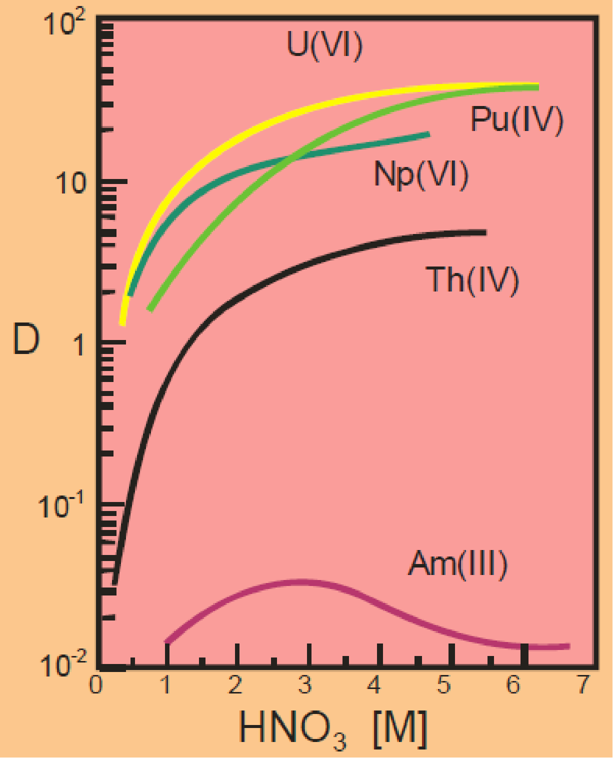
Solvent extraction has been an important separation technique since the early days of the Manhattan Project, when scientists extracted **$UO_2(NO_3)_2$** into **diethyl ether** to purify the uranium used in the first reactors.

In our days the SF reprocessing plants utilise PUREX process or extraction of (IV and VI valent) actinides with **tri-butyl phosphate (TBP) in kerosene**.



$Pu^{4+} + 4NO_3^- + 2TBP \leftrightarrow Pu(NO_3)_4 . 2TBP$

$UO_2^{2+} + 2NO_3^- + 2TBP \leftrightarrow UO_2(NO_3)_2 . 2TBP$



HDEHP (bis(2-ethylhexyl) phosphoric acid)-
extracts A(III) and Ln(III) quantitatively from HCl or HNO_3

Ion-exchange

Based on the **reversible exchange of ions between a solution and the resin**.

Ion-exchange resin- insoluble, inert polymeric matrix containing fixed charged groups (exchange sites) associated with mobile counter-ions of opposite charge, exchanged for ions from the solution.

The **exchange sites** are acid or base groups (amines, phenols, and carboxylic or sulfonic acids) used over a specific pH range where they are in their ionic form.

Typical functional groups for cation exchangers are **sulfonate $RSO_3^-H^+$ or carboxylate $RCOO^-H^+$**. The **quaternary-amine cation, $RNH_3^+Cl^-$** is a common exchange group for anion exchange resins.

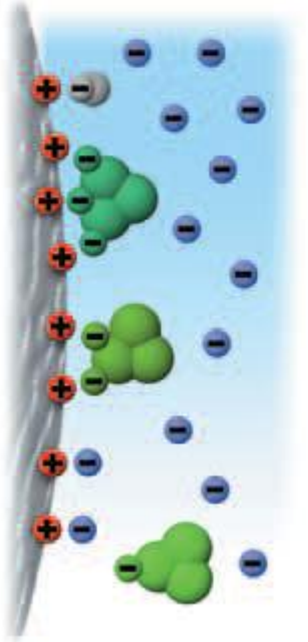
Ion-exchange

In a practical description of ion-exchange equilibria, the weight distribution coefficient K_d, and the separation factor, α, are significant.

The weight distribution coefficient is defined as:

$$K_d = \frac{[A_1]}{[A_2]}$$

where A_1 is the activity of the RN adsorbed on 1 g of the dry resin, and A_2 is the remaining RN activity in 1 mL of solution after equilibrium has been reached.



The separation factor refers to the ratio of the distribution coefficients for two ions that were determined under identical experimental conditions:

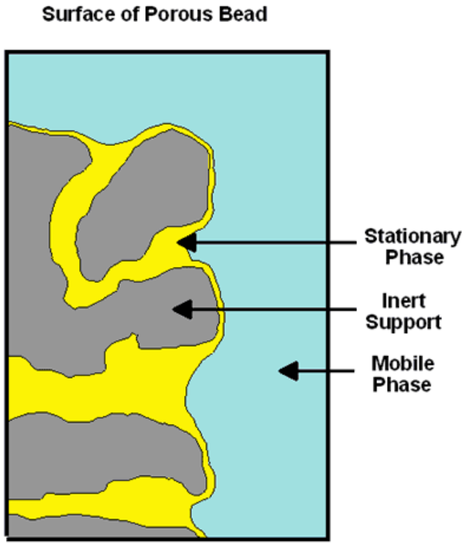
$$\alpha = \frac{[K_d, A]}{[K_d, B]}$$

where A and B refer to a pair of ions.

Extraction chromatography

Extraction chromatography combines the selectivity of liquid-liquid extraction with the rapidity of chromatographic methods.

The separation of the radionuclides is based on the distribution of RNS between an organic and an aqueous phase. The extractant is adsorbed on the surface of an inert support and corresponds to the organic phase.

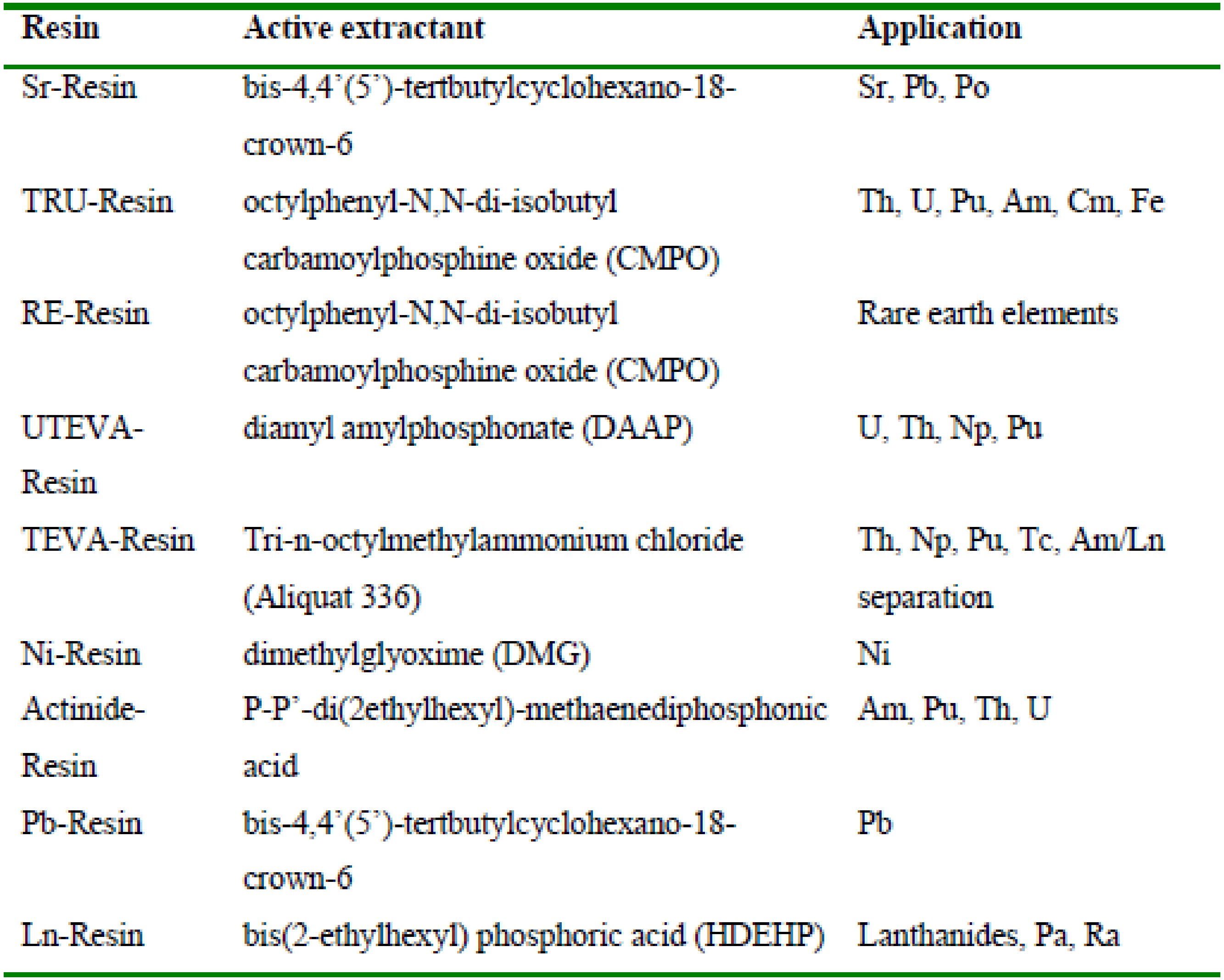


A wide variety of extractants is used :

- **Acidic extractants** (e.g. HDEDP) which exchange protons
- **Amines and ammonium salts** (e.g. Aliquat 336) which exchange simple anions against anionic complexes
- Organic molecules containing **P=O groups** (e.g. TBP) which exchange water molecules in the hydration sphere
- **Crown-ethers** which retain cations in function of their size

E.P. Horwitz et. al. Separation and preconcentration of actinides from acidic media by extraction chromatography. Analytica Chim Acta 281(2) p. 361-372 (1993)

Extraction chromatography

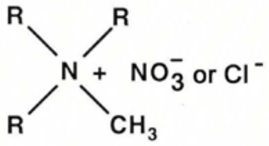


Resin	Active extractant	Application
Sr-Resin	bis-4,4'(5')-tertbutylcyclohexano-18-crown-6	Sr, Pb, Po
TRU-Resin	octylphenyl-N,N-di-isobutyl carbamoylphosphine oxide (CMPO)	Th, U, Pu, Am, Cm, Fe
RE-Resin	octylphenyl-N,N-di-isobutyl carbamoylphosphine oxide (CMPO)	Rare earth elements
UTEVA-Resin	diamyl amylphosphonate (DAAP)	U, Th, Np, Pu
TEVA-Resin	Tri-n-octylmethylammonium chloride (Aliquat 336)	Th, Np, Pu, Tc, Am/Ln separation
Ni-Resin	dimethylglyoxime (DMG)	Ni
Actinide-Resin	P-P'-di(2ethylhexyl)-methaenediphosphonic acid	Am, Pu, Th, U
Pb-Resin	bis-4,4'(5')-tertbutylcyclohexano-18-crown-6	Pb
Ln-Resin	bis(2-ethylhexyl) phosphoric acid (HDEHP)	Lanthanides, Pa, Ra

TEVA Resin

Figure 1

Trialkyl, methylammonium nitrate (or chloride)

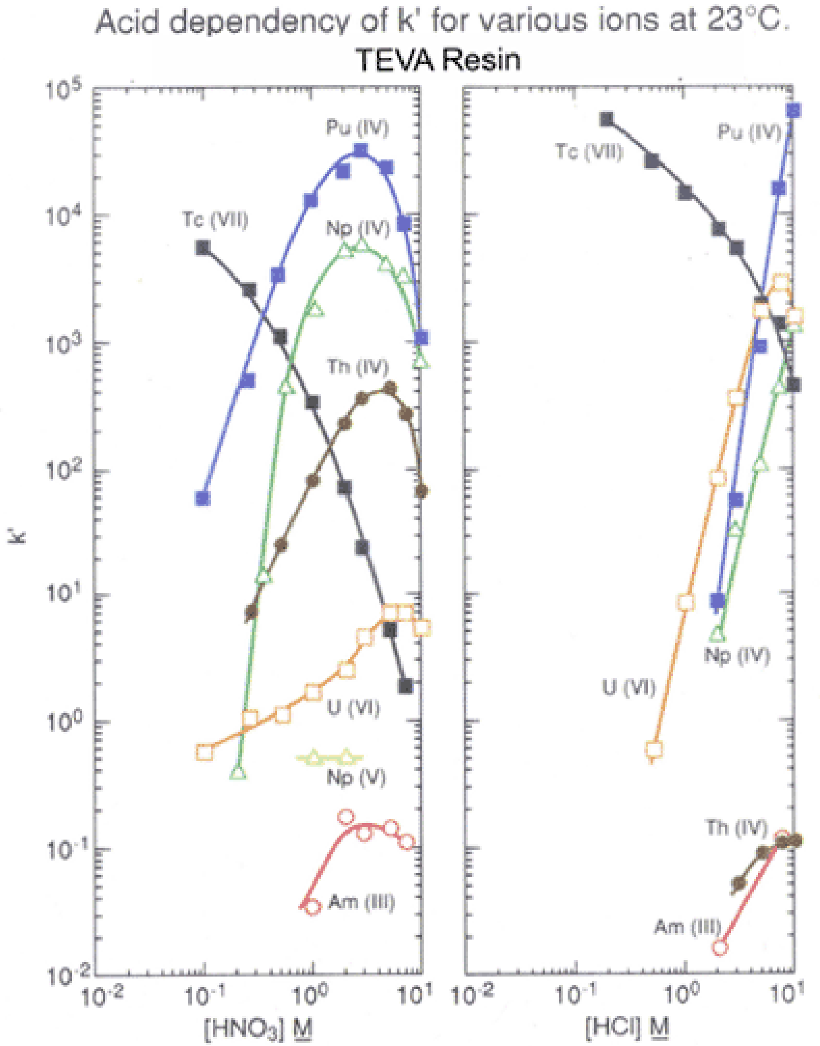


R = C_8H_{17} and $C_{10}H_{21}$

Active component- **aliphatic quaternary amine**.

The differences **between the uptakes for HNO_3 and HCl** can be exploited to separate certain actinides. I.e. all the tetravalent actinides can be loaded from 3M nitric acid and then by switching to 6M HCl, **Th(IV) can be selectively eluted**.

Figures 2 & 3


Acid dependency of k' for various ions at 23°C.

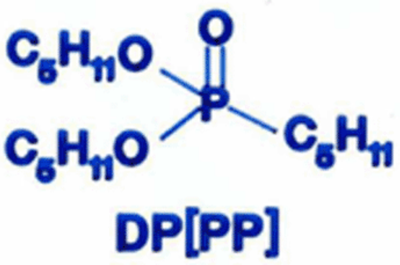
TEVA Resin

Horwitz, et al. (HP195)

UTEVA Resin

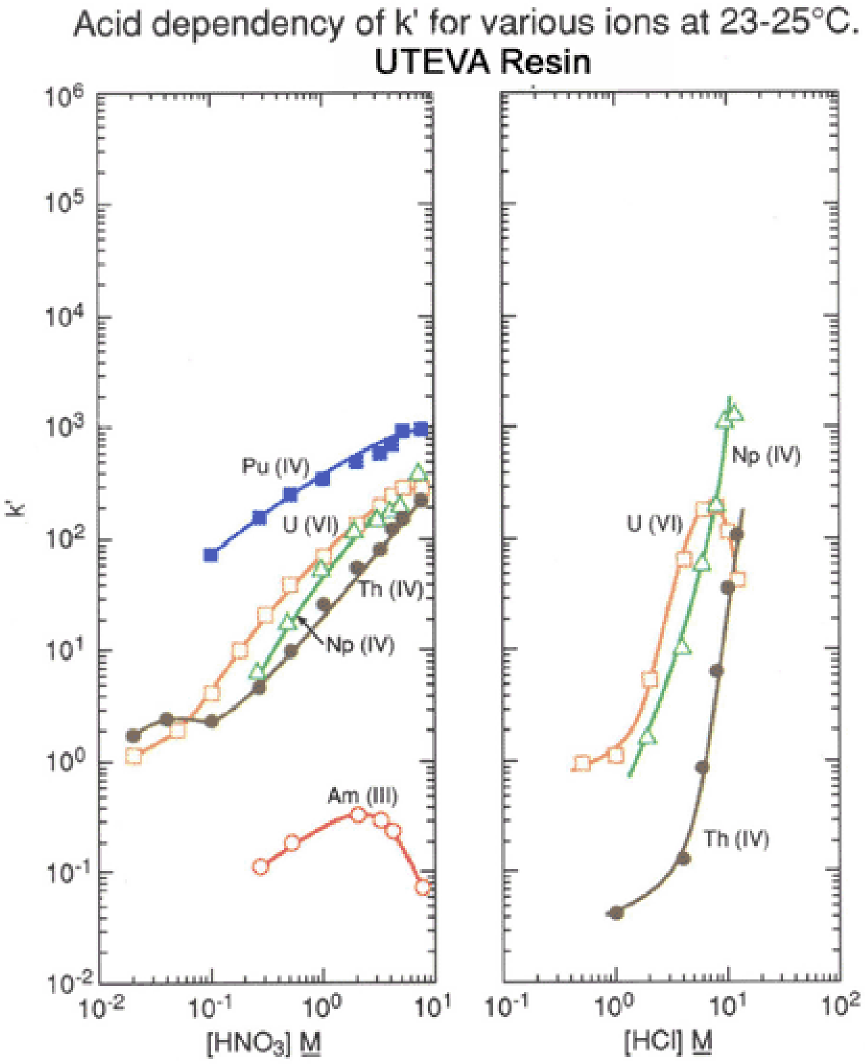
The extractant in the UTEVA Resin- diamyl, amylphosphonate DAAP

Figure 1



Dipentyl, pentylphosphonate
(or Diamyl, amylphosphonate – DAAP)

Figures 2 and 3


Acid dependency of k' for various ions at 23-25°C.
UTEVA Resin

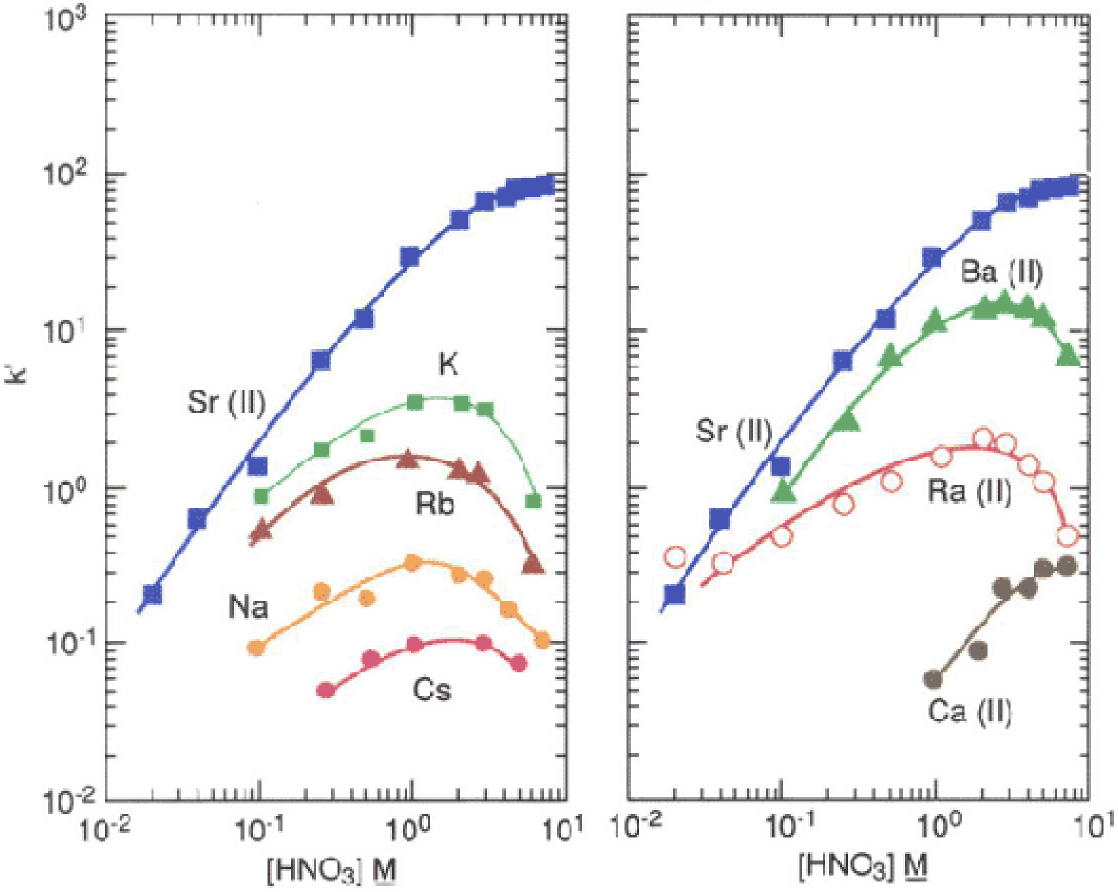
The large difference in Kd for U and Th in the range of 4-6M HCl allows **U/Th separation**.

Horwitz, et al. (HP392)

Sr Resin

Figures 2 and 3

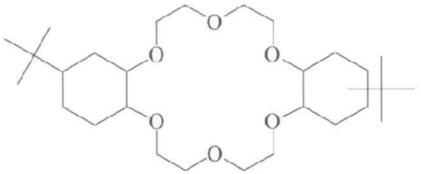
Acid dependency of k' for various ions at 23-25°C.
Sr Resin



Horwitz, et al., (HP292)

Figure 1

4,4'(5')-di-t-butylcyclohexano 18-crown-6



Diluent: 1-octanol

4,4'(5')-di-t-butylcyclohexano 18-crown-6 in 1-octanol

The uptake of **Sr increases at high [HNO_3]**. At 8M nitric acid, Kd is 10^2 and it falls below 0 at low [HNO_3].

Among the alkaline earth metals, **calcium has lowest uptake** and it is easy to separate Sr from Ca.

Ba retention is high, **but it falls off** at higher concentrations**.** To ensure adequate decontamination of Ba in Sr analysis, **load Sr on the resin from 8M nitric acid**, Ba is eluted, leaving a pure Sr fraction.

IAEA
International Atomic Energy Agency

Quantification of radionuclides

A typical laboratory may be equipped with the following radiometric instrumentation:

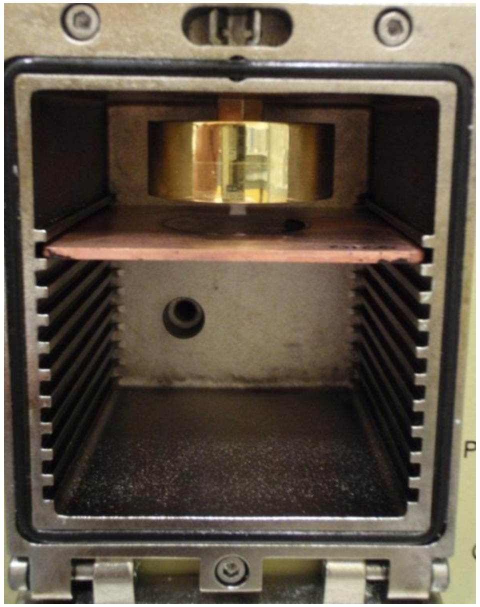
- **Gas proportional** detectors for alpha and beta-particle counting
- High resolution germanium **HPGe** detectors for gamma detection and spectrometry
- Solid-state detectors for **alpha spectrometry**
- Liquid scintillation counters suitable for both alpha- or beta-emitting radionuclides **(LSC)**
- Photon Electron Rejecting Alpha Liquid Scintillation **(PERALS)**

Quantification of radionuclides

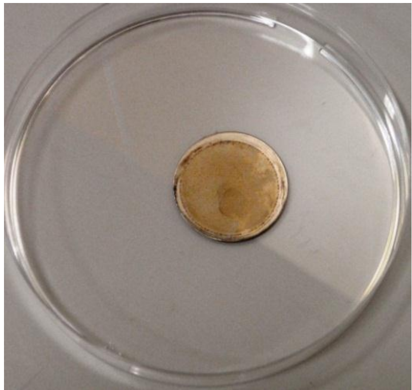
Some labs may also be equipped with atom and ion counting instrumentation:

- **Mass Spectrometric Analyses**
  - ✓ **Inductively Coupled Plasma-Mass Spectrometry (ICP-MS)**
  - ✓ **Thermal Ionization Mass Spectrometry (TIMS)**
  - ✓ **Accelerator Mass Spectrometry (AMS)**

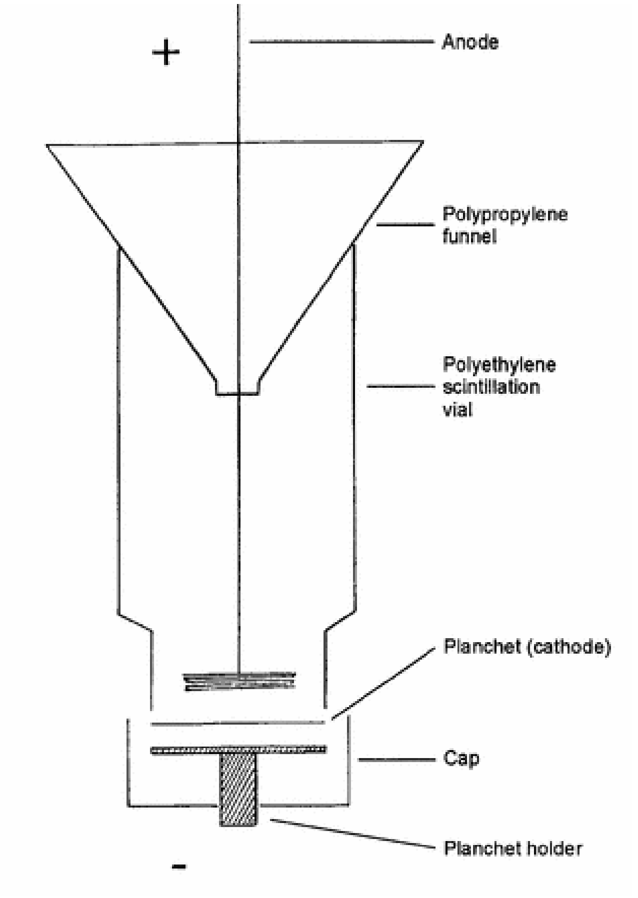
Alpha spectroscopy



- Solid state (PIPS) detectors
- Low background
- High counting efficiency
- Resolution 15-50 keV
- Energy calibration with calibration sources
- Counting efficiency measured using tracers (U232, Pu242, Am243, Po209 etc)
- Electroplating or micro precipitation for SP

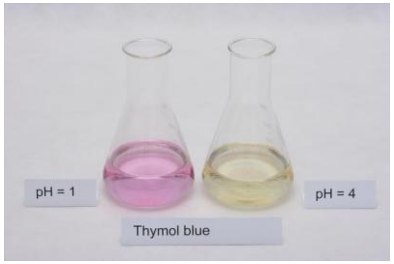


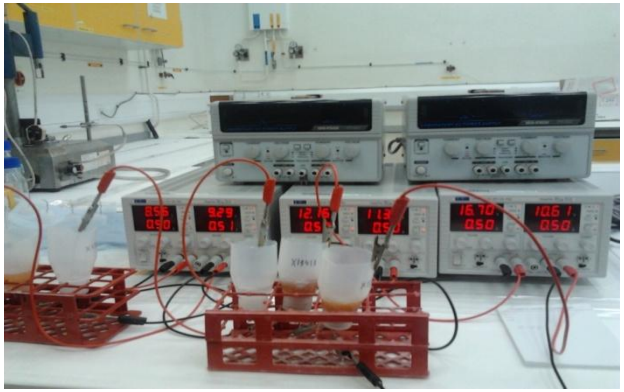
Electrodeposition



- Sample evaporation
- Re-dissolving in c.HNO_3
- Treatment with H_2O_2
- Evaporation
- Re-dissolving in 0.4 M H_2SO_4
- pH indicator (*thymol blue)*
- Add ammonia until reach light pink colour (pH=2)
- Transfer to ED cell
- Electroplate for 2 h at 0.5A
- Add excess of ammonia
- Electroplate for 5 more min

$(NH_4)_2SO_4$/
H_2SO_4
pH=2
E=0.5A
T=2h

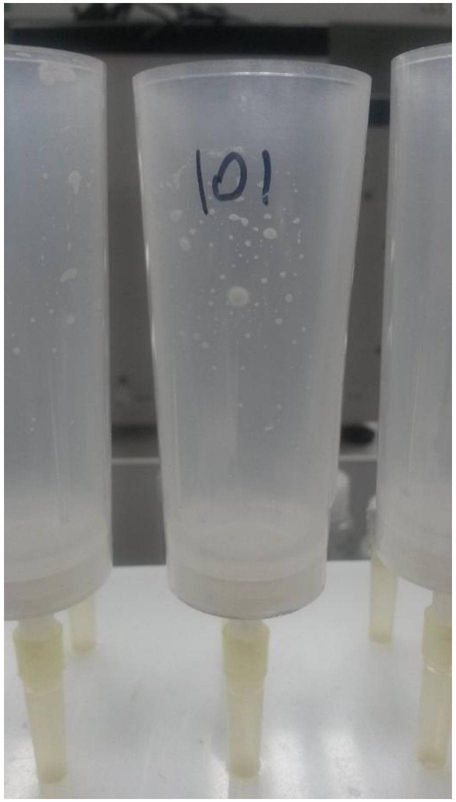




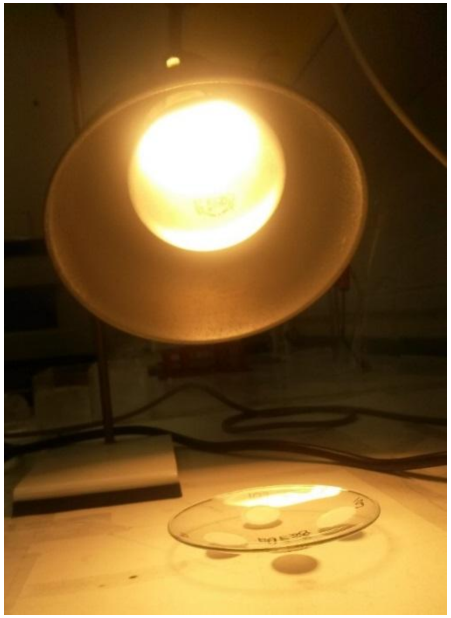
Micro-precipitation

Experimental set-up

Filtration step

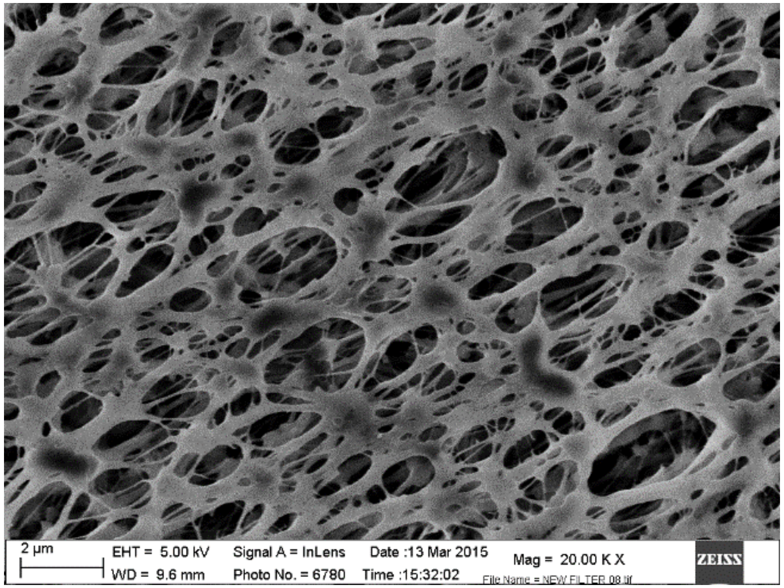


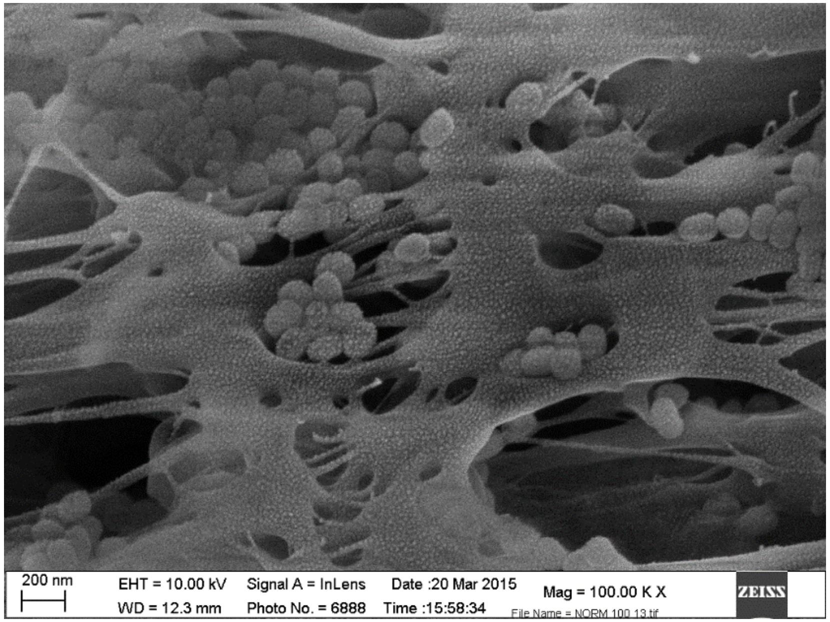
Residual precipitate (even after washing with ethanol)

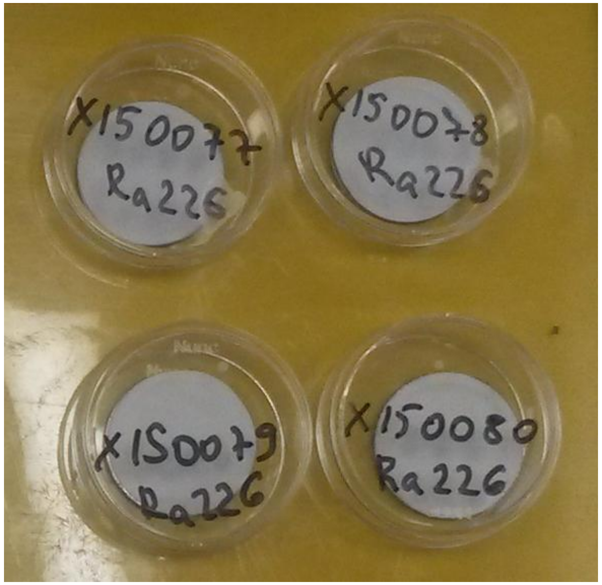


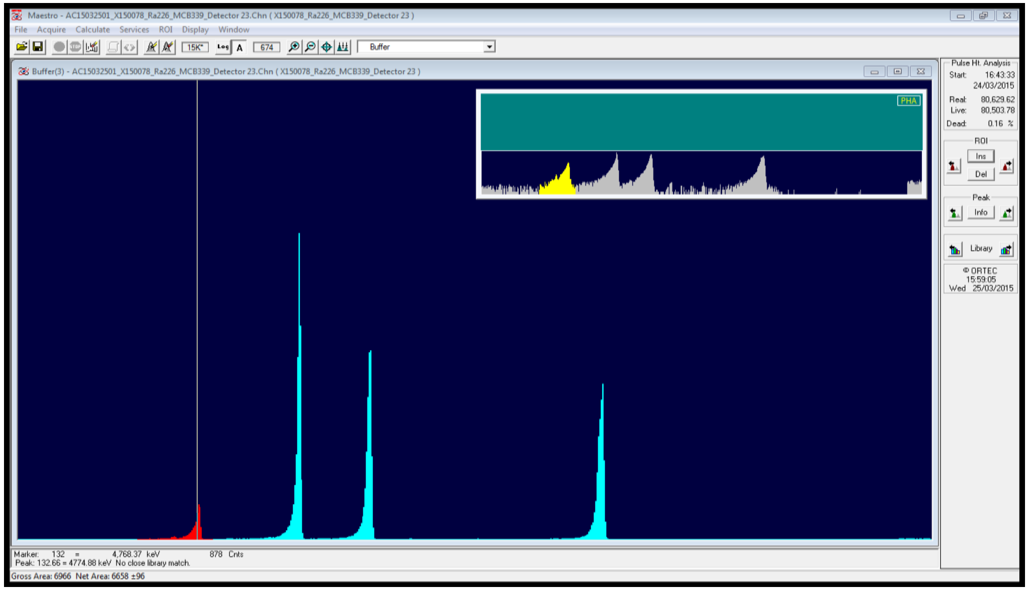
Drying step

Micro-precipitation









Maestro - AC15032501_X150078_Ra226_MCB339_Detector 23.Chn (X150078_Ra226_MCB339_Detector 23)

Buffer(3) - AC15032501_X150078_Ra226_MCB339_Detector 23.Chn (X150078_Ra226_MCB339_Detector 23)

Pulse Ht. Analysis
Start: 16:43:33
24/03/2015
Real: 80,629.62
Live: 80,503.78
Dead: 0.16 %

© ORTEC
15:59:05
Wed 25/03/2015

Marker: 132 = 4,768.37 keV 878 Cnts
Peak: 132.66 = 4774.88 keV No close library match.
Gross Area: 6966 Net Area: 6658 ±96

Examples of radioanalytical schemes – ^{nat}U, ^{232}Th

Adding tracers	i.e. ^{232}U, ^{229}Th
Sample dissolution	i.e. borate fusion
Separation	AG1, UTEVA
Counting	α-spec, mass spec

Examples of radioanalytical schemes – ^{226}Ra

- Direct γ-spectroscopy
- Pre-concentration with $BaSO_4$ followed by γ-spectroscopy
- Alpha-spectroscopy

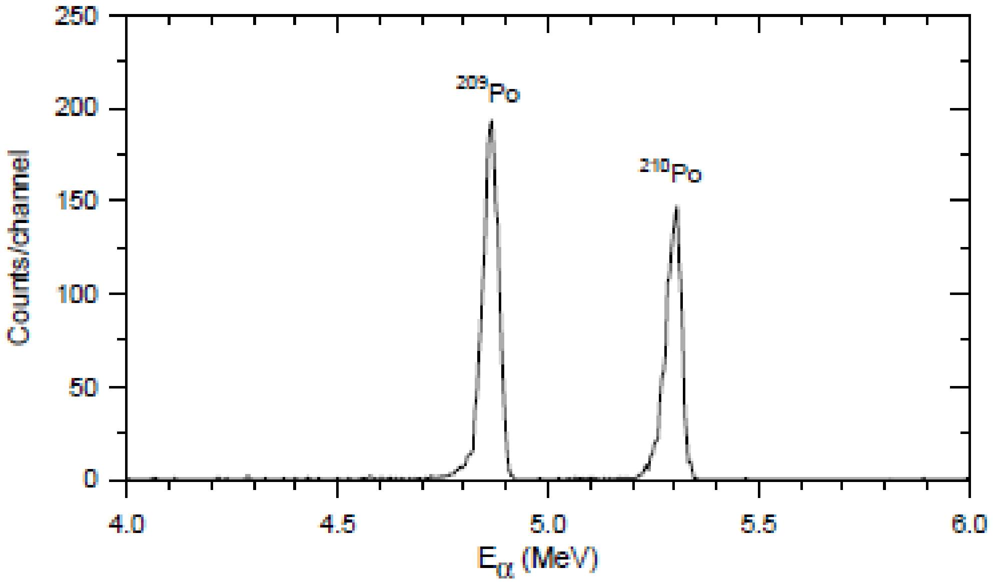
  -MnO_2 column

  -$BaSO_4$ micro-precipitation

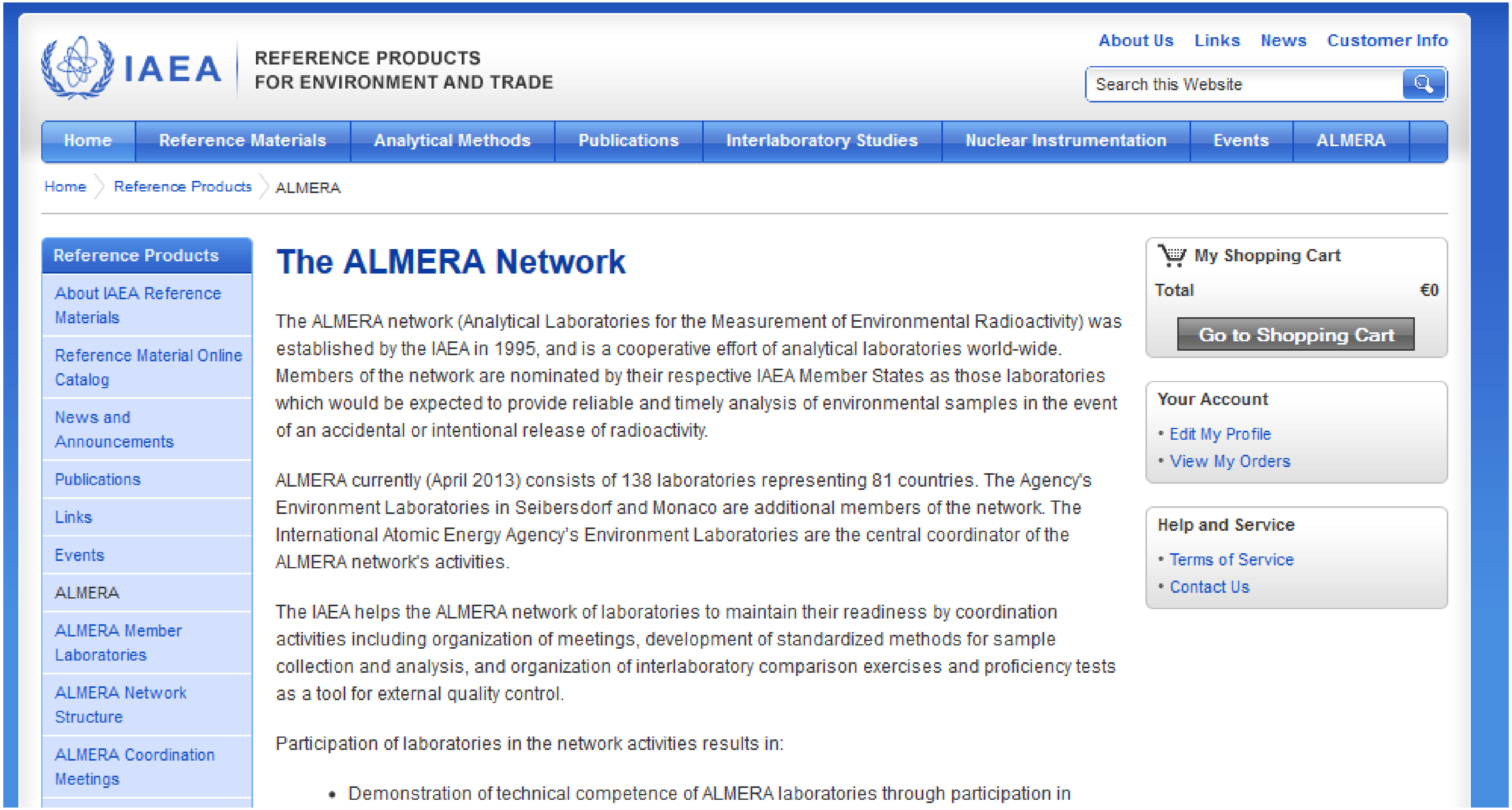
  -alpha-spec

Examples of radioanalytical schemes –^{210}Po

- Adding tracer ($^{208, 209}$Po)
- Separation on Sr resin/HCl
- Self deposition on Ag disc
- Alpha-spectroscopy



IAEA
International Atomic Energy Agency



The ALMERA Network

The ALMERA network (Analytical Laboratories for the Measurement of Environmental Radioactivity) was established by the IAEA in 1995, and is a cooperative effort of analytical laboratories world-wide. Members of the network are nominated by their respective IAEA Member States as those laboratories which would be expected to provide reliable and timely analysis of environmental samples in the event of an accidental or intentional release of radioactivity.

ALMERA currently (April 2013) consists of 138 laboratories representing 81 countries. The Agency's Environment Laboratories in Seibersdorf and Monaco are additional members of the network. The International Atomic Energy Agency's Environment Laboratories are the central coordinator of the ALMERA network's activities.

The IAEA helps the ALMERA network of laboratories to maintain their readiness by coordination activities including organization of meetings, development of standardized methods for sample collection and analysis, and organization of interlaboratory comparison exercises and proficiency tests as a tool for external quality control.

Participation of laboratories in the network activities results in:

- Demonstration of technical competence of ALMERA laboratories through participation in

IAEA
International Atomic Energy Agency

ALMERA validated methods

A procedure for the sequential determination of Polonium-210, Lead-210, Radium-226, Thorium and Uranium isotopes in phosphogypsum by liquid scintillation counting and alpha spectrometry

A procedure for the rapid determination of Radium-226 and Radium-228 in drinking water by prompt liquid scintillation counting

Determination of Po-210 in water samples by alpha spectrometry
Analytical Methodology for the Determination of Radium Isotopes in Environmental Samples

Thank you.
Any questions?

National Measurement System

The National Measurement System delivers world-class measurement science & technology through these organisations

NPL National Physical Laboratory

LGC Setting standards in analytical science

nel technology for life

National Measurement Office

The National Measurement System is the UK's national infrastructure of measurement laboratories, which deliver world-class measurement science and technology through four National Measurement Institutes (NMIs): LGC, NPL, the National Physical laboratory, TUV NEL the former National Engineering Laboratory, and the National Measurement Office (NMO).

NPL

To develop & disseminate UK's measurement standards, ensure they are internationally accepted

To provide multidisciplinary R&D and technical services for public and private sector

To provide knowledge transfer and advice to industry, government and academia

NPL: Technical breadth

- Acoustics
- Advanced Materials
- Air Quality
- Biometrics
- Biotechnology
- Corrosion
- Dimensional metrology
- Environmental measurement
- Lasers
- Mass and force
- Micro/Nanometrology
- Neutron measurements
- Photonics
- Photometry/Colour
- Pressure
- RF/Microwaves
- Radiation Dosimetry
- **Radioactivity**
- Radiometry
- Scientific Software
- Sensory metrology
- Statistics
- Surface analysis
- Thermal measurements
- Time & Frequency
- Electrical Standards

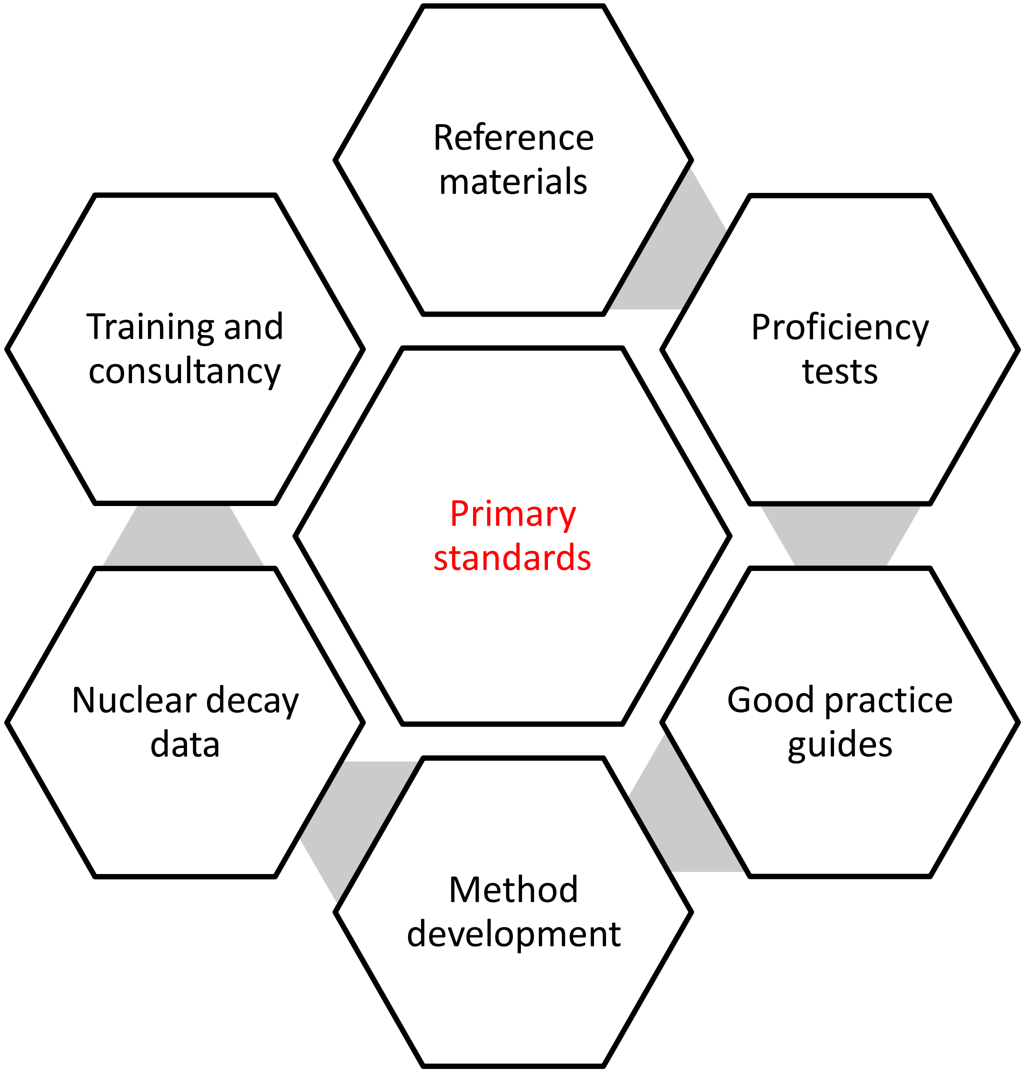
Mission of the radioactivity group

To enable users of radioactivity to measure radioactivity at an accuracy that is

fit for purpose

and traceable to international standards.

In practice



Reference materials

Training and consultancy

Proficiency tests

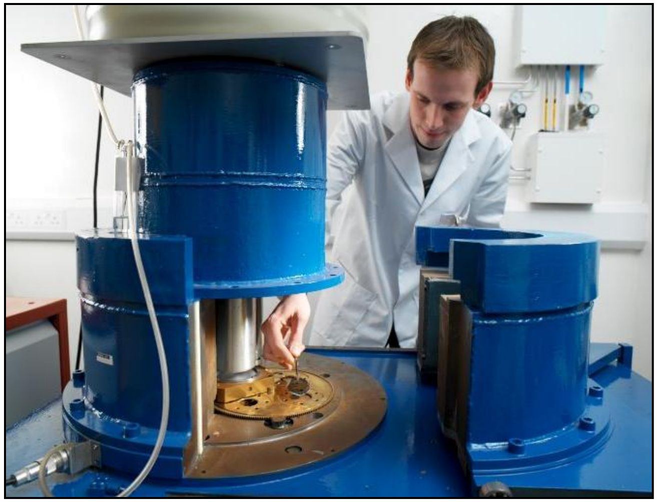
Primary standards

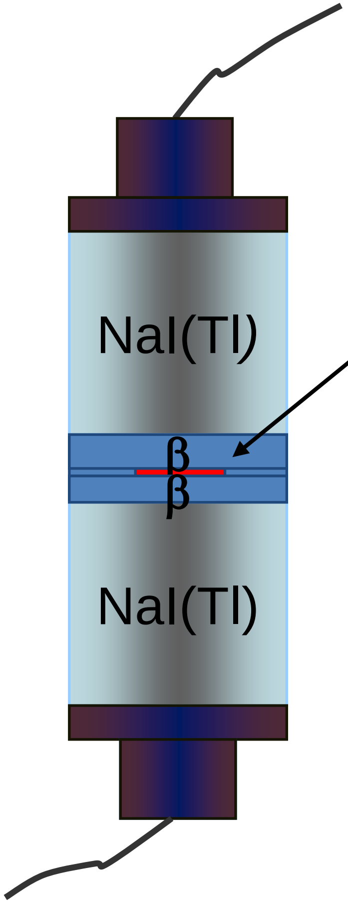
Nuclear decay data

Good practice guides

Method development

Radioactivity standards: Primary techniques (e.g. 4π β/γ coincidence counting)





Radioactive sample

$$A = \frac{N_\beta N_\gamma}{N_c}$$

Radioactivity standards: Secondary techniques

e.g.

Ionisation chambers

HPGe γ-spectrometers

Why Focus on Measurement

Measurement matters!

- The Environmental Permitting Regulations
- Site licence conditions
- WAC
- NICoP
- DQO

All require measurement of the radioactivity content of

- discharges
- waste materials

Why Focus on Measurement

- **Measurement matters!**

Improves environmental safety

Improves quality control, accuracy and repeatability

Fewer errors, reduced risk of prosecution

Reduced costs through accurate sentencing

Role of the international measurement system

To enable measurements that are:

- Fit for purpose
- Consistent between countries
- Independent of commercial and other pressures

How the international measurement system works

- **Primary standards** (of radioactivity, for example) are developed at National Measurement Institutes (NMIs)

- NMIs **cross-check the primary standards** against each other

- Each NMI **sets up methods** to enable other organisations to use the primary standards

R&D priorities

- Rapid, on-site, measurement techniques for more difficult samples
- New calibration materials to match the solid samples
- New measurement techniques

Mobile Laboratory

Aim: to set up a demonstration mobile laboratory for use on decommissioning projects, to analyse samples on site

- Delays in despatch and analysis of samples are delaying decommissioning projects
- New technology and regulatory changes make **rapid, on-site, measurements** feasible
- Loughborough University and NPL are setting up a **demonstration laboratory**
- The aim is to be the world's first **comprehensive mobile radiochemistry service**

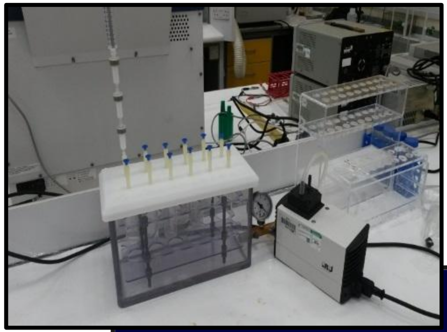
Loughborough University

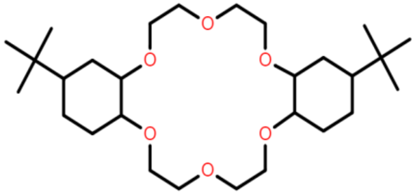
Mobile Laboratory

The aim of this project is:

- To develop **innovative methods for the characterisation** of potentially radioactive waste materials
- Can be deployed onto nuclear and non-nuclear sites, thus providing **in-situ analysis.**

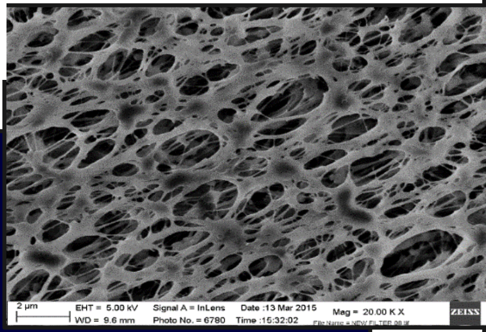
Extraction chromatography

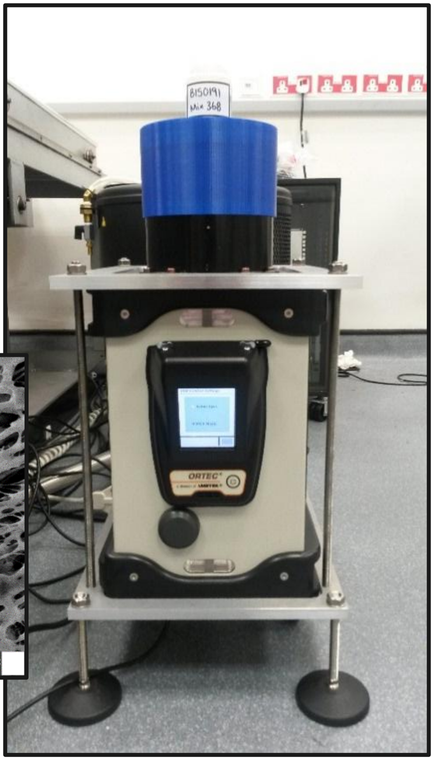




Crown-ethers

Resolve filters



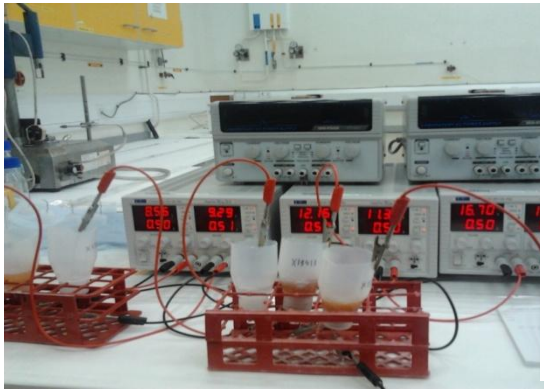


Portable HPGe γ-detection

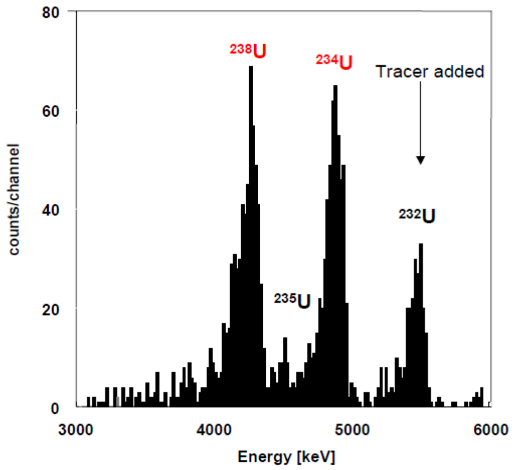
Mobile Laboratory

The aim of this project is:

- Recent developments in **digital signal processing,** detector technology and **radiochemistry** allow **rapid measurements** and expert interpretation of results.
- This service would **address a bottleneck** in nuclear decommissioning projects which can be delayed by several months while waiting for results from off-site laboratories.



electroplating

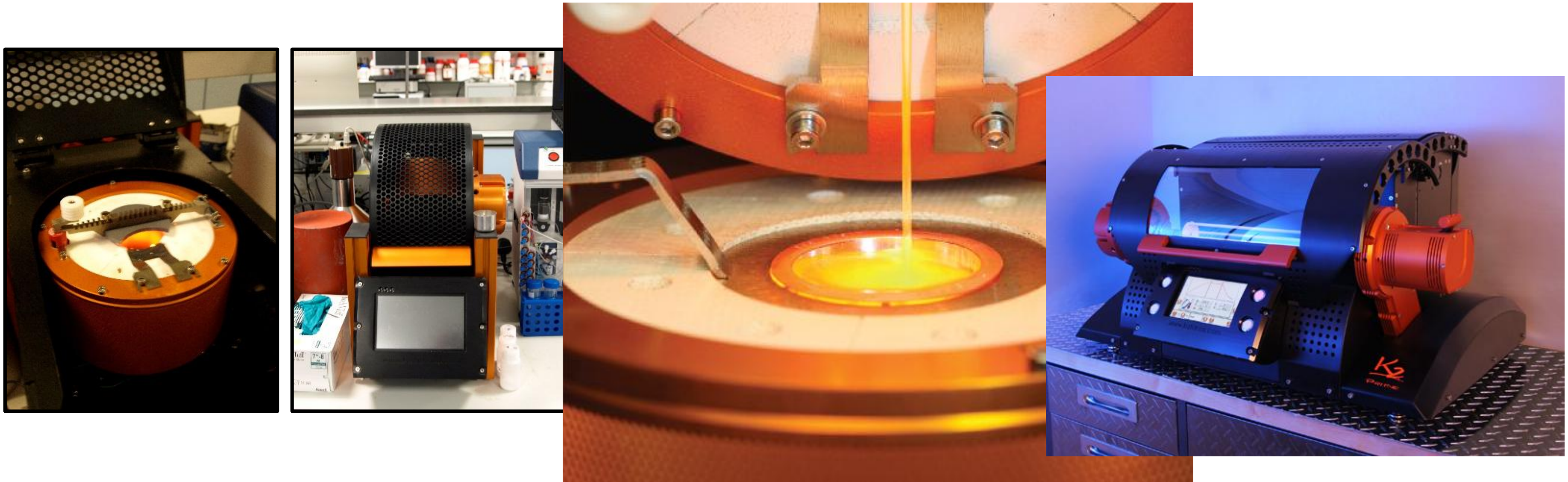


portable α-spec

Mobile lab equipment- PERALS & ARSIIe

Solid sample treatment (soil/sand/concrete)

- For the precise analysis of α- and β-emitters in solid samples **dissolution is required**
- Samples digested by **lithium borate fusion** using portable Katanax device
- Resulting **melt dissolved in acidic** solutions
- **Silicates removed** by PET precipitation
- Radiochemical separation and **analysis of each individual radionuclide fraction**



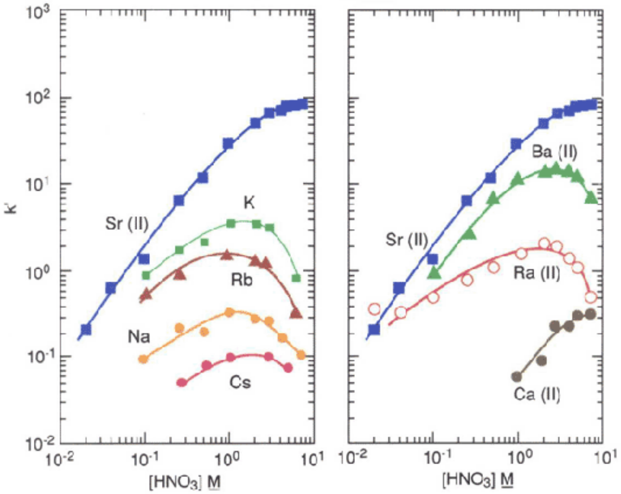
Radiochemistry separation of DTM radionuclides

- **Radiochemistry procedures** for the separation of **α- and β-emitting** nuclides by **rapid** state of the art methods
- Separations based **on ion exchange, solvent extraction and extraction chromatography**
- Rapid separations using **Vacuum box and stack cartridges** techniques

Figures 2 and 3

Acid dependency of k' for various ions at 23-25°C.

Sr Resin

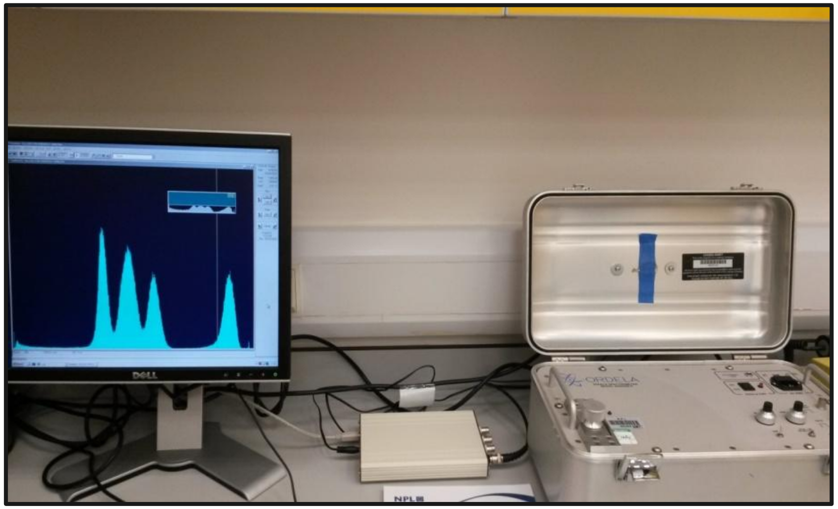


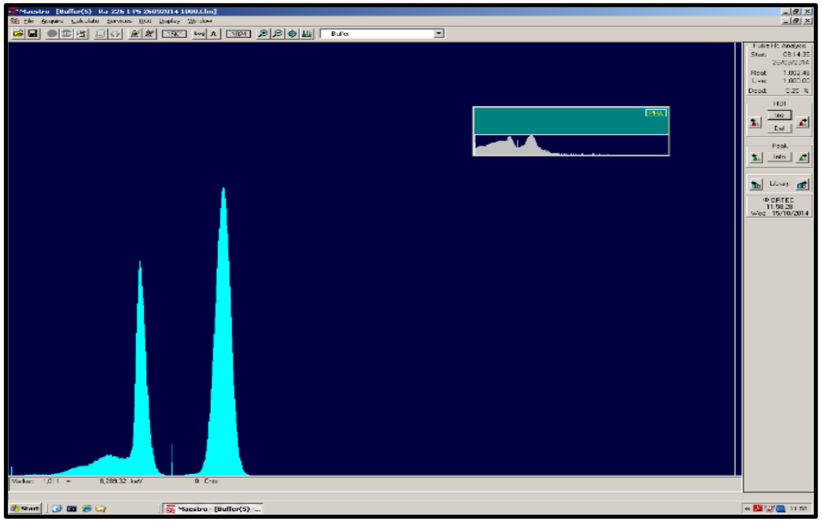
Horwitz, et al., (HP292)

PERALS

(Photon Electron Rejecting Alpha Liquid Scintillation spectrometry)

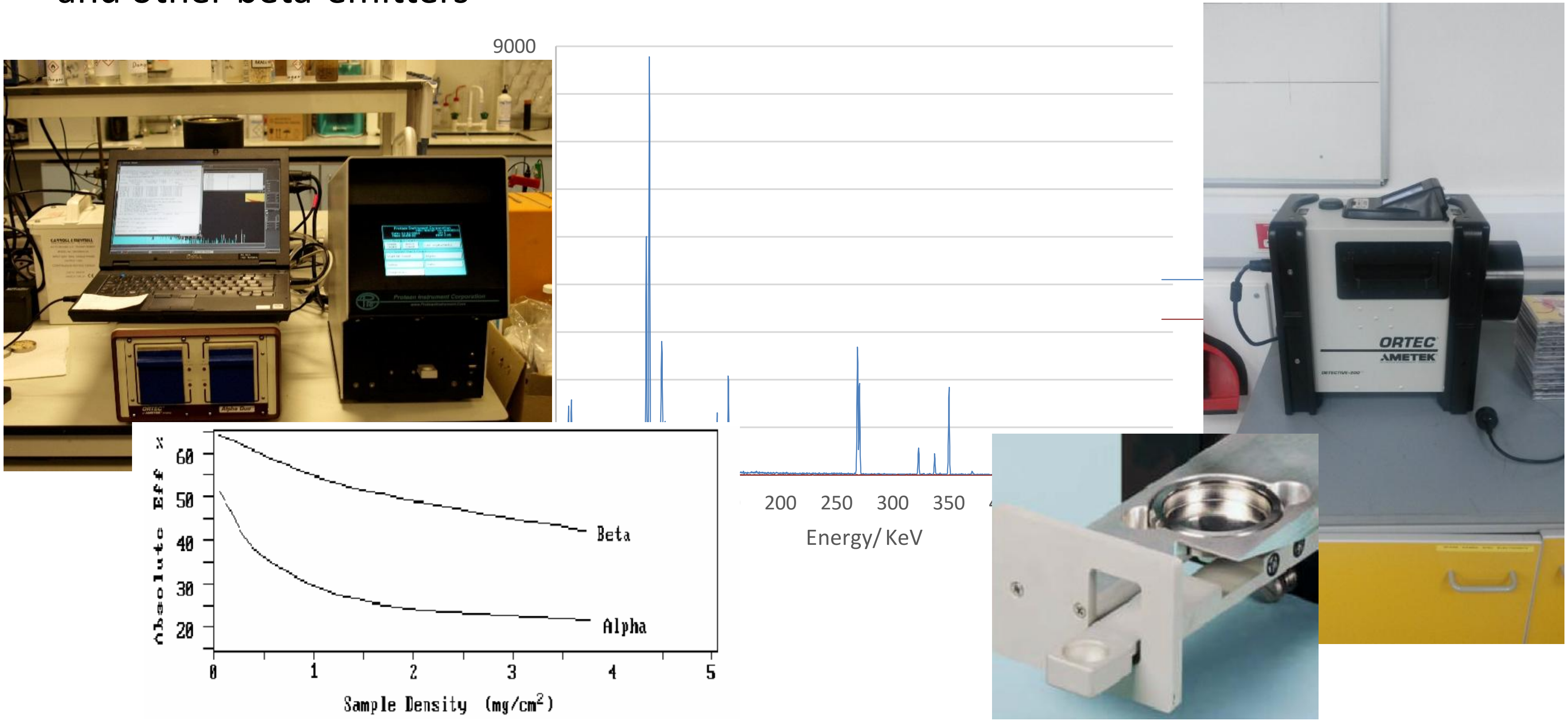
- Designed **to quickly screen soil, water, liquid, air filter, and smear samples** for alpha contamination by rapid alpha spectrometry
- Features **counting efficiency** as high as **99.7 %**
- Based on **extractive LS cocktails**, combines radionuclide separation with LS counting
- Suitable for the determination of total alpha activity, **U, Th, Ra, Sr analysis**





Radiometric counting

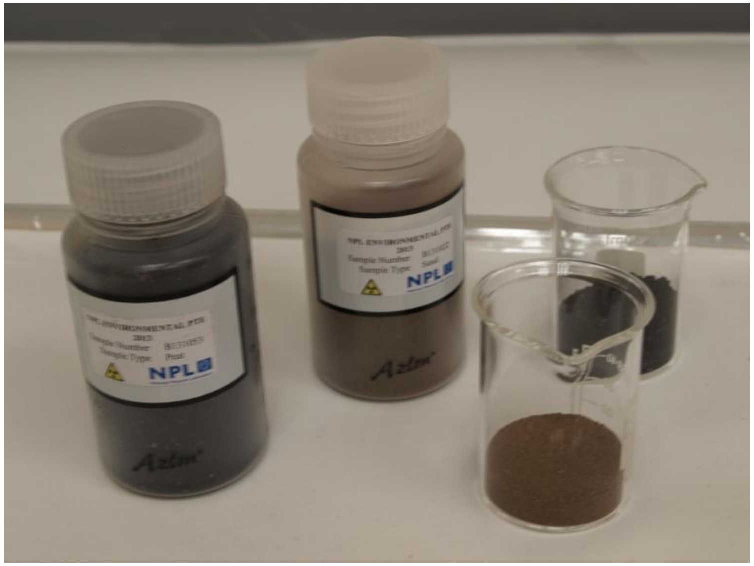
- **Portable** high-resolution electrically cooled **HPGe γ-detector** (Detective 200TM), battery/AC/DC powered, highly stable digital electronics, designed for **on-site analysis** of wide range of γ-emitting radionuclides
- **Gross α/β counter** (MPC900), suitable for measuring the total activity of actinides, Sr90 and other beta-emitters



Reference materials, proficiency tests; associated traceability

Reference materials

In addition to standard test methods and guides **that promote public and worker safety and environmental health, quality assurance, and regulatory compliance** within the nuclear industry, it is important to have **standard reference materials** that provide data on the physical and chemical properties of waste and waste forms.



Reference materials, proficiency tests; associated traceability

Inter-laboratories collaboration and proficiency tests

The **reliability of the final result at the end of the characterization process cannot be evaluated in a simple way**. The **uncertainty** of this result is a **function** of its **precision** (repeatability, reproducibility) and **accuracy** (difference from the real value, **expressed as the difference between a result (or mean) and the true value**).

The **precision** of the result can be determined by an **internal control** in the laboratory using **certified standards**. However, the determination of the **accuracy requires** the following specific actions:

- Carrying out **repeated analysis using different methodology**, different analysts and different techniques,
- Carrying out **control analysis with a reference matrix**,
- Participation in **inter-laboratory comparisons** (proficiency testing)

Inter-laboratory comparison studies can be a reliable way to determine the precision and accuracy of the results.

Reference materials, proficiency tests; associated traceability

An intensive national and international collaboration among scientists, and the **exchange of information** on waste samples and **radio-analytical methods** among the **different laboratories/institutions** can help to identify the scope of application, the strengths and weaknesses of the methods and provide **opportunities for improvement**, harmonization, validation and finally qualification of selected methods within the scientific community.