Materials radiopurity and radioactive assays

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SOUP 20|21: INFN School on Underground Physics

OBJECTIVES of this lecture

After this class, the student should be able to:

- Know the most important **sources of natural radioactivity**
- Understand the principles of **equilibrium and disequilibrium** in the decay chains
- Grasp the principles of the **cosmogenic** production of radionuclides
- Understand the general framework of the problems posed by the **Rn-222**
- Figure out the importance of **material assays and radiopurity** related aspects for low-background techniques
- Know the principle of the most used **techniques** for the assays

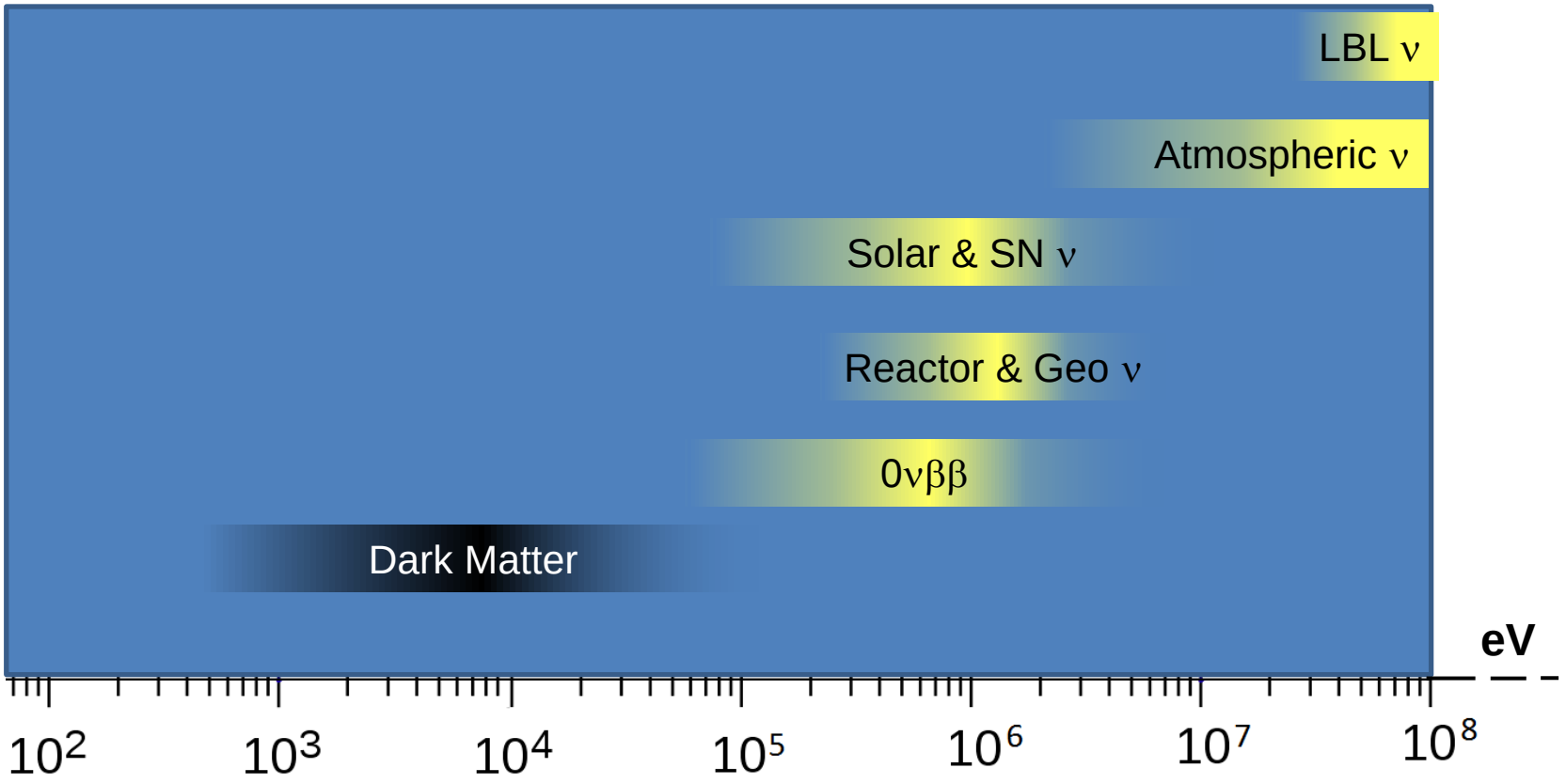
What you are supposed to know:

- Principles of radioactive decay
- Principles of nuclear radiation
- Principles of interaction of radiation with matter
- General aspects related to the rare-events searches and low-background techniques

Literature

- Heusser "*Low-Radioactivity Background Techniques*"
  Ann. Rev. Nucl. Part. Sci. 45: 543 (1995).
- Formaggio J. A., Martoff C. J., "*Backgrounds to sensitive experiments underground*", Ann. Rev. Nucl. Part. Sci. 54, 361-412 (2004).
- S. Cebrian, "*Cosmogenic Activation in Double Beta Decay Experiments*", Universe 6 (2020) 10, 162 .
- M. Wojcik, G. Zuzel, "*Review of high-sensitivity Radon studies*", *Int.J.Mod.Phys.A* 32 (2017) 30, 1743004.
- M. F. L'Annunziata, "*Handbook of Radioactivity Analysis*", Elsevier.
- G.R. Gilmore, "*Practical gamma-ray spectrometry by Gordon Gilmore*", John Wiley & Sons.
- Several with *material assay* papers from different low background experiments (XENON, EXO, LZ, GERDA, NEXT...) .

Searching for solar neutrinos, double beta decays, or dark matter particles



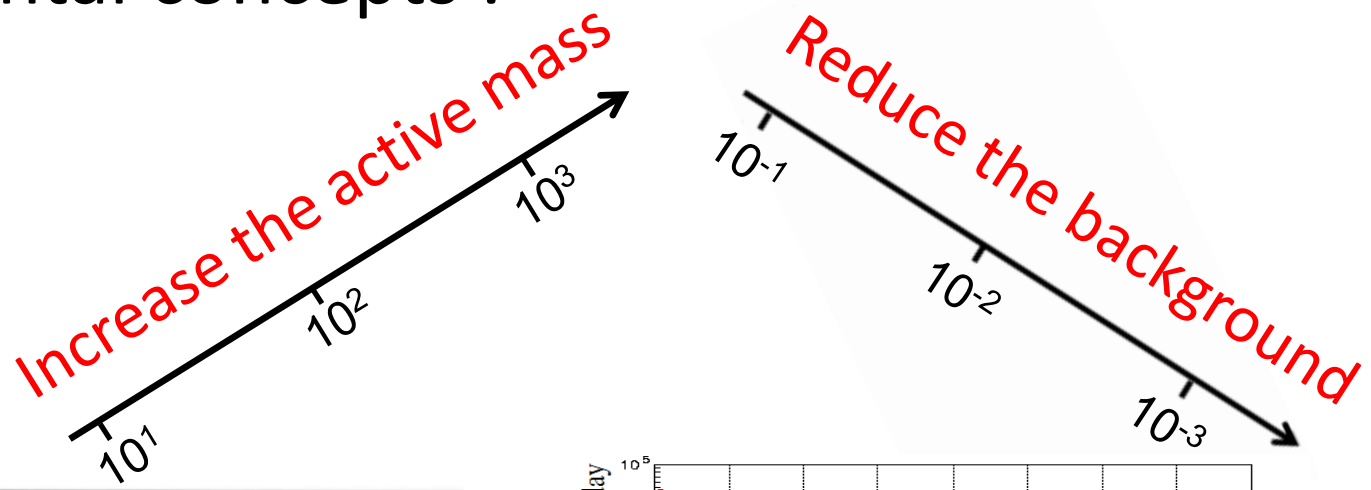
Searching for solar neutrinos, double beta decays, or dark matter particles

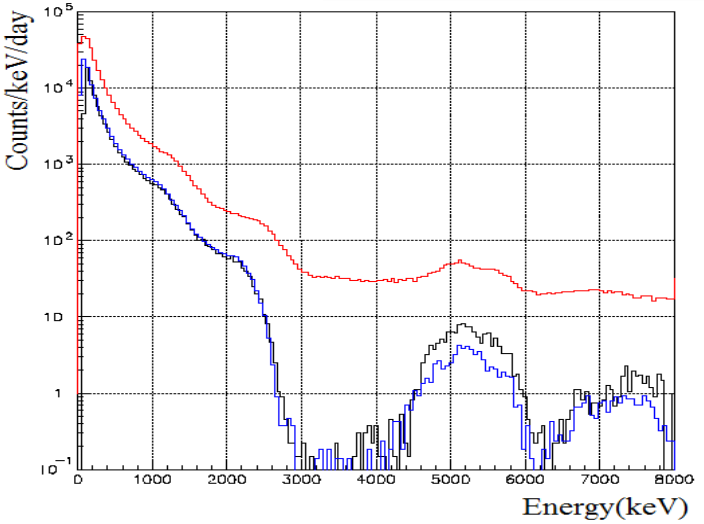
Event rates as low as a few per year are predicted

- *Improved technologies*
- *Optimized detector design*
- *Better techniques able to filter a weak signal out of background and noise*
- *Data mining, machine learning….etc*

Searching for solar neutrinos, double beta decays, or dark matter particles

Two fundamental concepts :





Uninteresting (and spurious) signals

- Although they vary from experiment to experiment:
  - cosmic rays
  - environmental radioactivity
  - from airborne activity (radon)…
  - **…. and from intrinsic contamination of the detector**
- It must be precisely understood and minimized before the detector is constructed

Setting the stage: general aspects concerning the materials background

- α's, β's, γ's, … from the materials surrounding the active volume produce the ultimate background for the UG experiments

- It is very difficult to reduce this type of background
  - Limited im… …elds, rock
- A simple and e… …y (to some extent) is to minimize … …ed the build the detector

- The **radio-purit**… …y is **crucial** for the sensitivity that … …re-event searches

https://me.me/i/this-seems-likea-decent-amountof-pasta-what-the-okay-im-19124040

Material radiopurity: general aspects

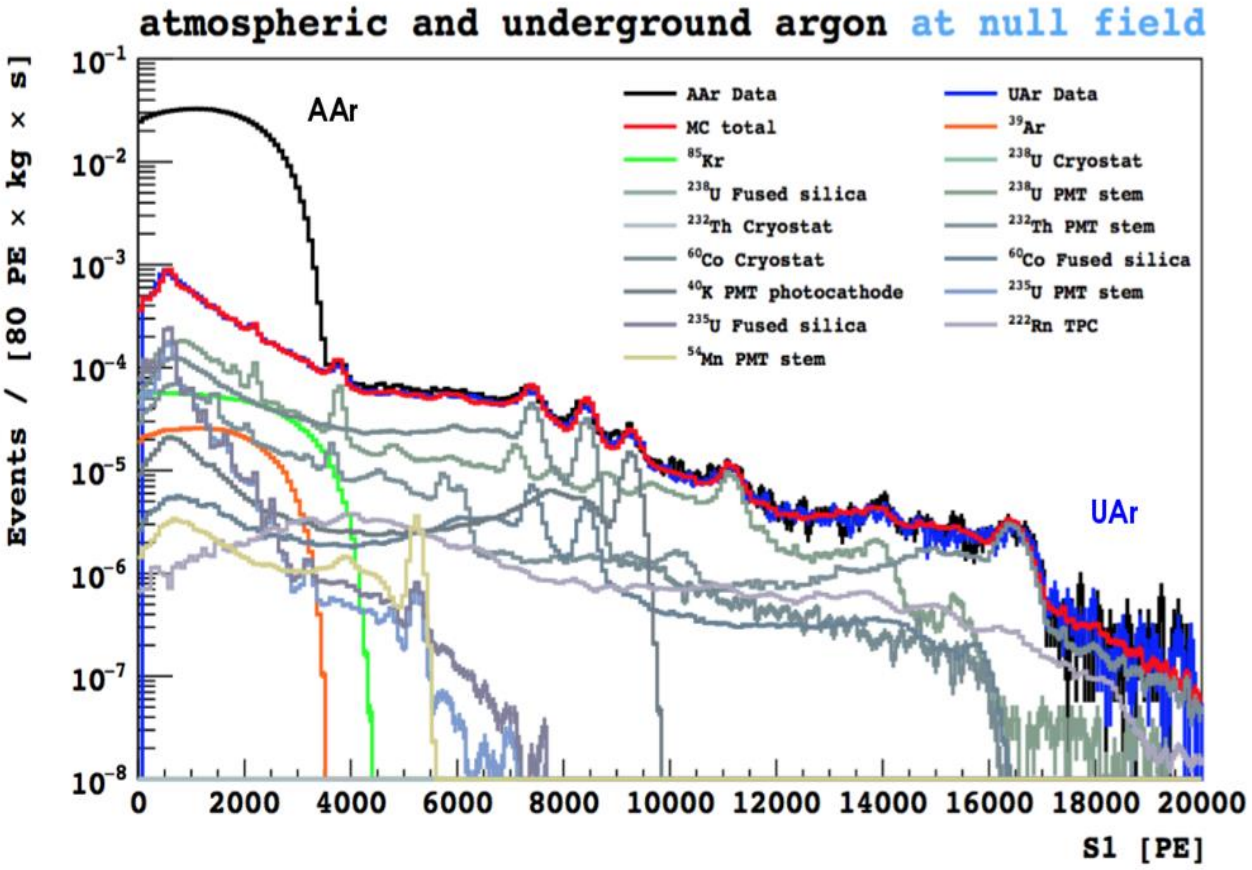
- The radio-contamination of the material cannot be 0

The raw material may have been contaminated while in the ground or the material itself may be made of an element that has radio-isotopes

- Over time, a great interest in developing experimental techniques suitable to improve, verify and maintain the radiopurity of these detectors has arisen: nowadays It is a frontier aspect for rare-event searches

Material radiopurity: general aspects

DS-50 @ LNGS



Cryostat, Sensors, support structure…Ar

Strategy

- **Dedicated measurements to ensure the cleanliness of the materials used in the detector's construction: quantify their content of radioactive impurities.**

- **Multiple counting methods/facilities: gamma counting, chemistry, radio-chemistry…**

Sample, vendor,….

- The level of radiopurity measured in samples of commercial raw materials from different providers can vary by orders of magnitude.

- The contamination can be different, even in batches of the same material with different geological or chemical history because of differences in chemical composition of ores.

… batch, manufacturing, storage

- Identify the vendor, secure individual production batches
- Radiopurity of manufactured items can be significantly different from the one measured in samples of raw materials
- Clean production: the materials contaminated during manufacturing or synthesis by intentional or unintentional addition of radio-contaminated, catalysts, mold, lubricants, etc
- Avoid recontamination, stockpiling of clean materials in suitable storage

Art?

"Selecting candidate low-radioactivity materials is as much an art as it is a science"

(*Nucl.Instrum.Meth.A* 839 (2016) 6-11)

- It relies on inference from previous measurements and on the experience of the experimentalist
- Critical task: the testing and certification of candidate materials involves precision measurements that consume **significant amounts of time, cost and an enormous effort**
- Candidates must be selected judiciously: how well this can be done depends upon the quality of information available to the researcher
- Of particular importance is their access to previous measurements of similar materials

What we have to measure?

How can we measure it?

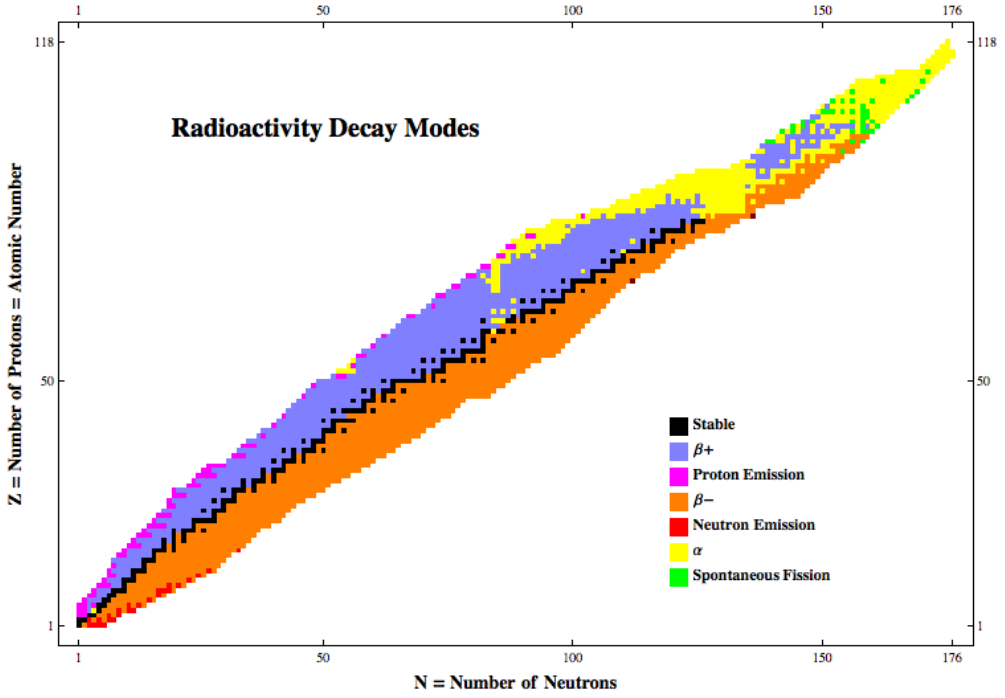
What we have to measure?

How can we measure it?

Radionuclides

https://www-nds.iaea.org/relnsd/vcharthtml/VChartHTML.html

- The number of neutrons is about equal to the number of protons. (“shell model” of the nucleus: nuclear energy levels similar to the electron shells)
- N=Z up to atomic number Ca-20.
  For heavier stable atoms, the number of neutrons increases faster than the number of protons
- *Above Z* = 82 (or 83), no stable isotopes of the elements discovered so-far



N of parent atoms decreases *exponentially* with time

$$N = N_0 e^{-t/\tau}$$

λ is the *decay constant* of the particular atom species in the sample → A = N λ = N/τ
half-life $t_{1/2}$ of a radioactive sample is the time required for half of the atoms in the sample to decay

$$t_{1/2} = (\ln 2)/\lambda = 0.693 \cdot \tau$$

Raconv

http://wwwae.ciemat.es/raconv/raconv.php

RACONV, the activity conversion program (version 3.31)

© Roberto Santorelli (C-code), Pablo García (PHP) - CIEMAT

Element: 137Cs e.g. 226Ra - Chain option: one of 238Uc, 235Uc, 232Thc (case matters)
Amount: 1 float, e.g. 100
Density: float, e.g. 0.5 (default 1)
Unit: g Activity: Bq, Ci, g, N (number of atoms), m and k multiples allowed - Contamination: ppb, g/g, mBq/kg

Enviar consulta Reset

```
Your input :      1 g 137Cs
Value :           1
Units :           g
Nuclide :         137Cs
Density factor :  1

Chain calculation : No
        RESULTS
Nuclide:  137Cs
Raconv ID: 3
Fraction:  100.000000

Nb. Atoms :          4.398710e+21
Mass number :        136.907000
Mass :               1.000000e+00 g[137Cs]
Nb. of moles:        7.304228e-03

Half life :          3.007000e+01 y (1.098283e+04 days)
Mean lifetime :      4.338184e+01 y
Decay constant :     2.305112e-02 1/y (7.304615e-10 1/s)
Specific activity :  3.213088e+12 Bq/g[137Cs] (8.684014e+07 µCi/
```

```
        ABSOLUTE ACTIVITY:
Activity : 3.213088e+15 mBq (8.684014e+10 nCi)
Activity : 3.213088e+12 Bq  (8.684014e+07 µCi)
Activity : 3.213088e+09 kBq (8.684014e+04 mCi)

        CONTAMINATION if input activity is per g of the sample:
Contamination (/g) : 1.000000e+00 g[137Cs]/g
Contamination (/g) : 1.000000e+09 ppb        (1.000000e+12 ppt)
Contamination (/g) : 3.213088e+12 Bq/g       (8.684014e+07 µCi/g)
Contamination (/g) : 3.213088e+18 mBq/kg     (8.684014e+16 pCi/kg)
        CONTAMINATION if activity is per kg of the sample:
Contamination (/kg) : 1.000000e-03 g[137Cs]/g
Contamination (/kg) : 1.000000e+06 ppb        (1.000000e+09 ppt)
Contamination (/kg) : 3.213088e+09 Bq/g       (8.684014e+04 µCi/g)
Contamination (/kg) : 3.213088e+15 mBq/kg     (8.684014e+13 pCi/kg)

        SUMMARY

Your input : 1 g 137Cs

ID      NUCL      Nb. Atoms          Ac[Bq]          Mass[g]      BR alpha
3       137Cs     4.398710e+21       3.213088e+12    1.000000e+00
```

http://nucleardata.nuclear.lu.se

WWW Table of Radioactive Isotopes

$^{137}_{55}Cs_{82}$

Half life:	30.07 y *3*
Jp:	7/2+
S_n (keV):	8278.3 *19*
S_p (keV):	7416 *7*
Prod. mode:	Fission product Fast neutron activation Thermal neutron activation
ENSDF citation:	NDS 72,355 (1994)
Literature cut-off date:	1-Oct-1993
Author(s):	J.K. Tuli
References since cut-off:	^{137}Cs decay from 1993-98 (NSR)

Decay properties:

Mode	Branching (%)	Q-value (keV)
b^-	100	1175.63 *17*

Data sets:

Mode	Data set name	Display data
b^-	137CS B- DECAY	
	Tables:	Levels Gammas Betas
	ENSDF data:	Data
	Java applets:	Level scheme Beta spectrum

Gammas from ^{137}Cs (30.07 y *3*)

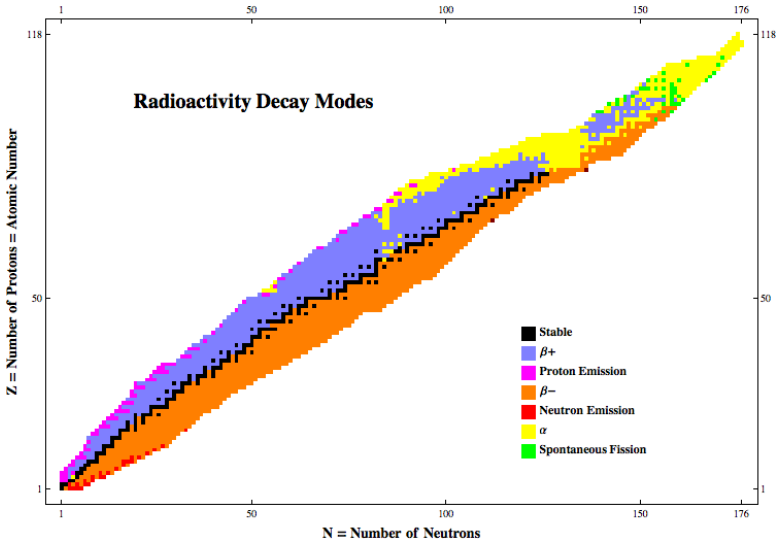
Eg (keV)	Ig (%)	Decay mode
283.53 *4*	0.00058 *8*	b^-
661.657 *3*	85.1 *2*	b^-

X-rays from ^{137}Cs (30.07 y *3*)

E (keV)	I (%)	Assignment
3,954	0.0143 *19*	Ba L_l
4,331	0.0064 *7*	Ba L_h
4,451	0.040 *4*	Ba L_{a2}
4,466	0.36 *4*	Ba L_{a1}
4,827	0.226 *23*	Ba L_{b1}
4,852	0.023 *5*	Ba L_{b4}
4,927	0.039 *8*	Ba L_{b3}
4,994	0.0030 *3*	Ba L_{b6}
5,156	0.074 *8*	Ba L_{b2}
5,531	0.033 *3*	Ba L_{g1}
5,797	0.0065 *14*	Ba L_{g2}
5,809	0.0093 *19*	Ba L_{g3}
31,452	0.000263 *8*	Ba K_{a3}
31,817	2.04 *5*	Ba K_{a2}
32,194	3.76 *8*	Ba K_{a1}
36,304	0.352 *8*	Ba K_{b3}
36,378	0.680 *15*	Ba K_{b1}
36,652	0.0079 *3*	Ba K_{b5}
37,255	0.215 *5*	Ba K_{b2}
37,349	0.0481 *20*	Ba K_{b4}

Betas from ^{137}Cs (30.07 y *3*)

Eb endpoint (keV)	Ib (%)	Decay mode
513,97	94.4 *2*	b^-
892,22	0.00058 *8*	b^-
1175,63	5.6 *2*	b^-



Isotopes on Earth are have been formed in processes of nucleosynthesis, either in the Big Bang, or in generations of stars that preceded the formation of the solar system

Natural occurring radionuclides

- **Primordial**

  found on the Earth that have existed in their current form since before Earth was formed

- **Cosmogenic**

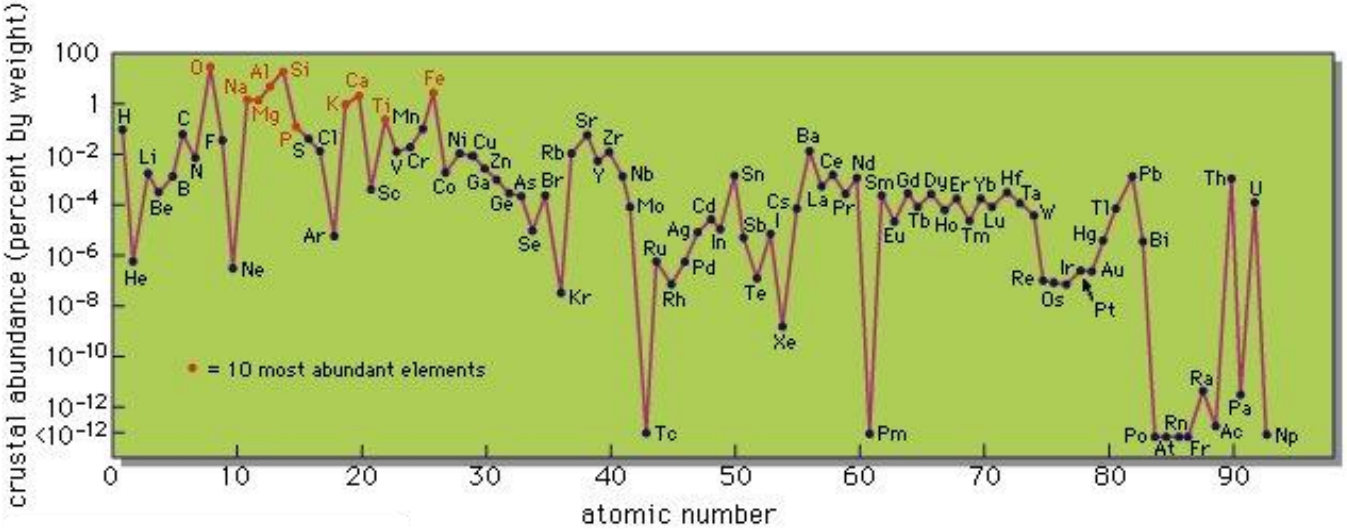
  Production of unstable nuclei by cosmic rays entering the Earth's atmosphere

Artificial radionuclides

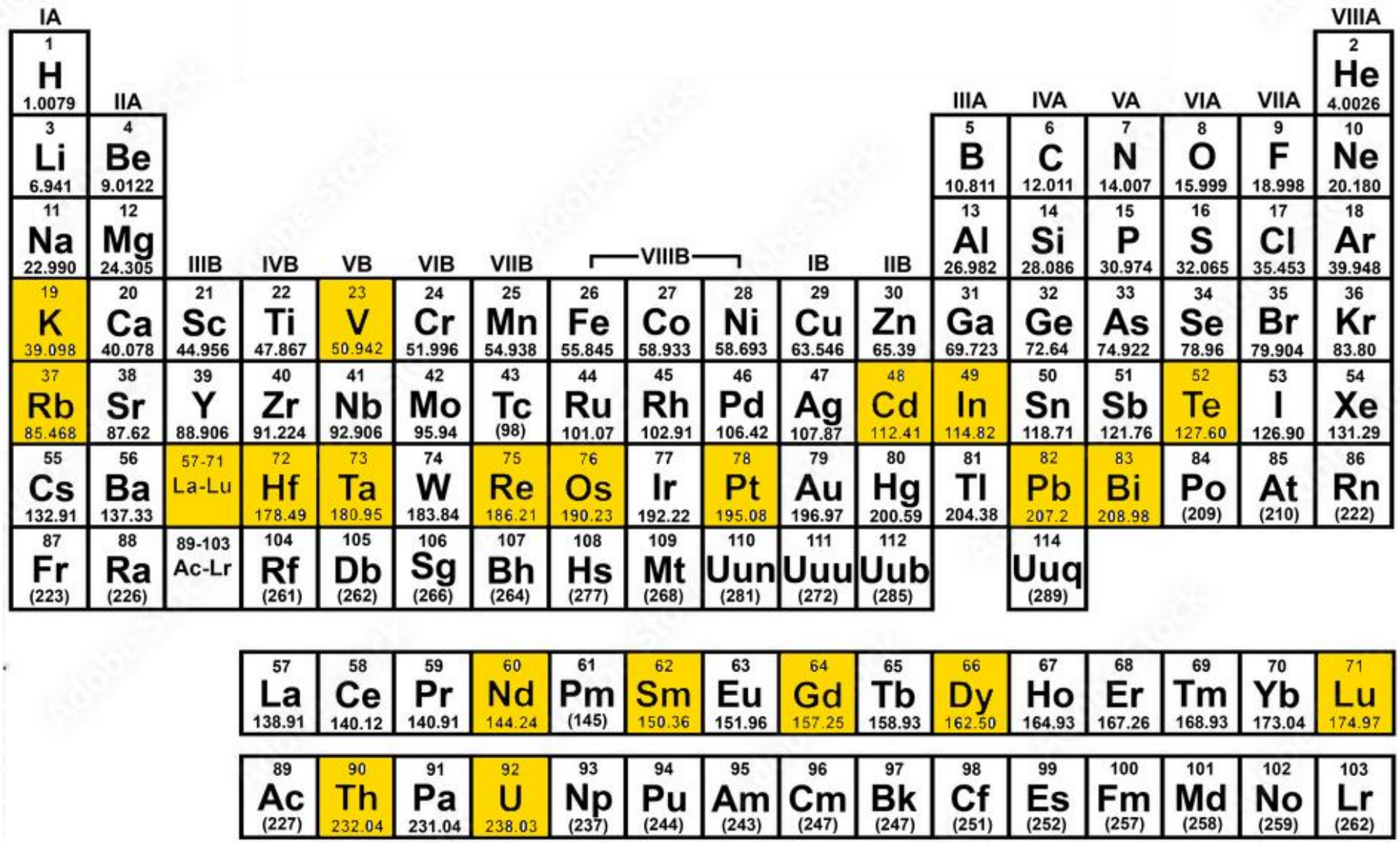
- **Anthropogenic** (nuclear tests, nuclear waste, accelerators…)

Primordial radionuclides

Abundance of the chemical elements in the Earth's upper continental crust



- There are 252 stable nuclides
- On Earth, $\sim 4.5 \times 10^9$ yr after its formation, most of the original unstable nuclei have decayed leaving only those with $\boldsymbol{t_{1/2} > 10^8}$ **yr**
- ≈34 radionuclides that have half-lives long enough to have survived from the formation of the Earth 4.5×10^9 years (isotopes of 28 separate elements)
- These long-lived nuclei involve either highly forbidden β-decays (large spin changes) or are α-decays that happen to have Q-values that place the half-lives in this range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2 He 4.0026
3 Li 6.941
4 Be 9.0122
5 B 10.811
6 C 12.011
7 N 14.007
8 O 15.999
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14 Si 28.086
15 P 30.974
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22 Ti 47.867
23 V 50.942
24 Cr 51.996
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27 Co 58.933
28 Ni 58.693
29 Cu 63.546
30 Zn 65.39
31 Ga 69.723
32 Ge 72.64
33 As 74.922
34 Se 78.96
35 Br 79.904
36 Kr 83.80
37 Rb 85.468
38 Sr 87.62
39 Y 88.906
40 Zr 91.224
41 Nb 92.906
42 Mo 95.94
43 Tc (98)
44 Ru 101.07
45 Rh 102.91
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47 Ag 107.87
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49 In 114.82
50 Sn 118.71
51 Sb 121.76
52 Te 127.60
53 I 126.90
54 Xe 131.29
55 Cs 132.91
56 Ba 137.33
57-71 La-Lu
72 Hf 178.49
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74 W 183.84
75 Re 186.21
76 Os 190.23
77 Ir 192.22
78 Pt 195.08
79 Au 196.97
80 Hg 200.59
81 Tl 204.38
82 Pb 207.2
83 Bi 208.98
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57 La 138.91
58 Ce 140.12
59 Pr 140.91
60 Nd 144.24
61 Pm (145)
62 Sm 150.36
63 Eu 151.96
64 Gd 157.25
65 Tb 158.93
66 Dy 162.50
67 Ho 164.93
68 Er 167.26
69 Tm 168.93
70 Yb 173.04
71 Lu 174.97
89 Ac (227)
90 Th 232.04
91 Pa 231.04
92 U 238.03
93 Np (237)
94 Pu (244)
95 Am (243)
96 Cm (247)
97 Bk (247)
98 Cf (251)
99 Es (252)
100 Fm (257)
101 Md (258)
102 No (259)
103 Lr (262)

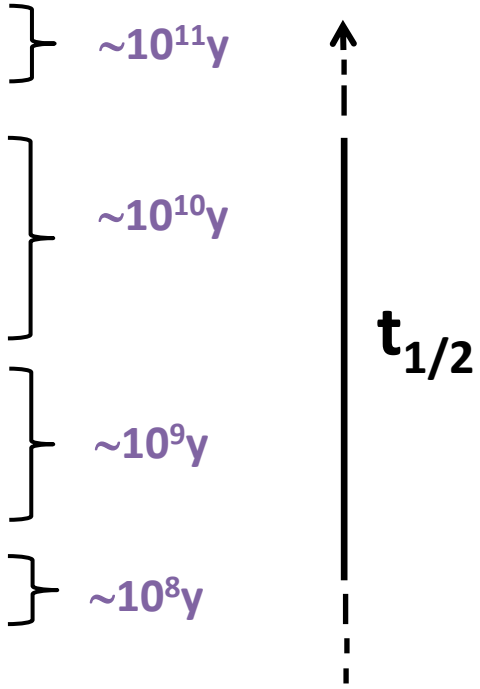
Relevant primordial radionuclides

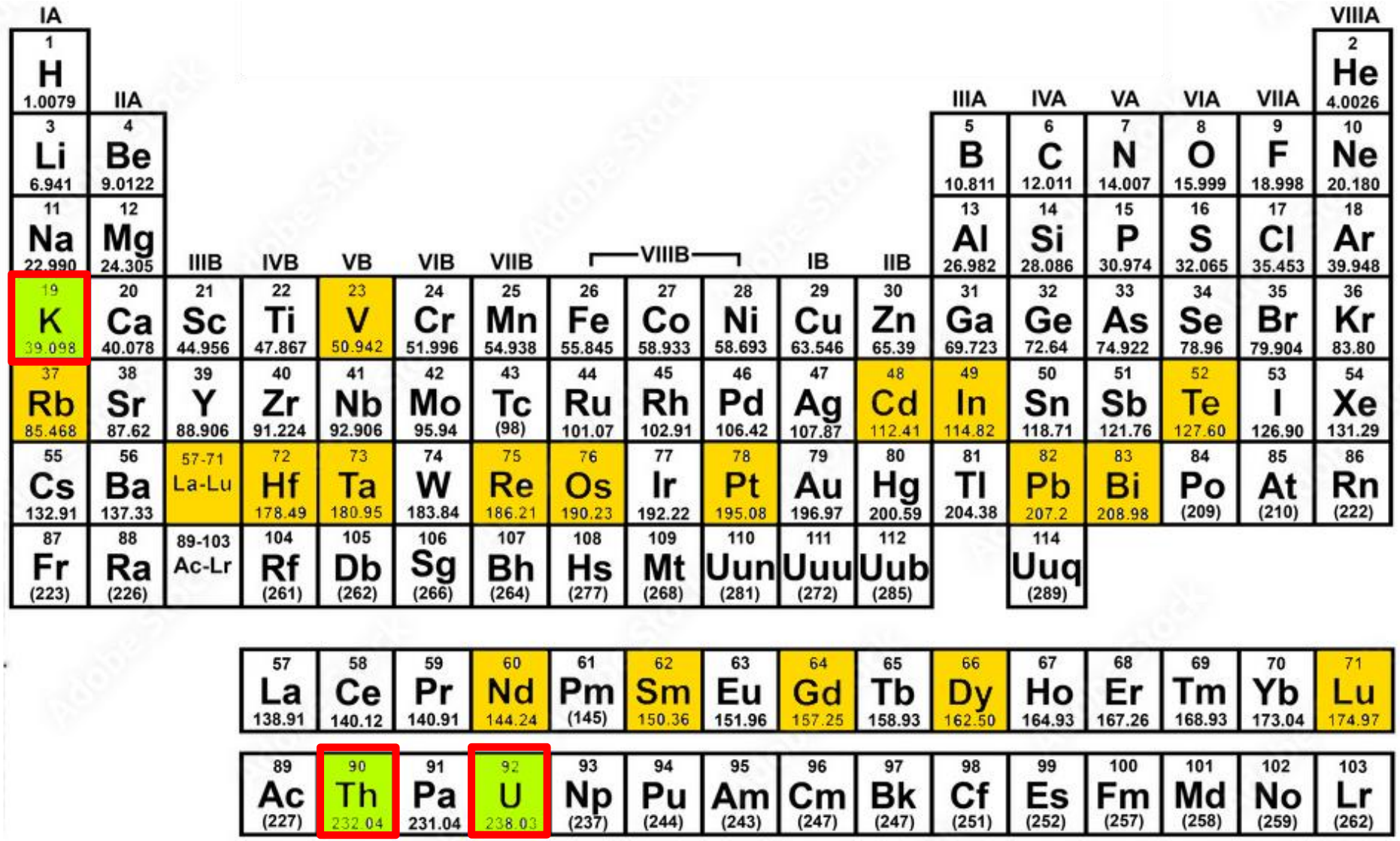
Most of them have half-lives significantly larger than the age of the Universe (they are practically stable with an abundance similar to the one of the stable isotopes of their respective elements)

There are only a handful of nuclides with half-lives comparable to the age of the earth

decay	half-life (years)	isotopic abundance (percent)	activity ($Bq\,kg^{-1}$) (element)	activity ($Bq\,kg^{-1}$) (crust)
$^{147}Sm \rightarrow {}^{143}Nd\,\alpha$	1.06×10^{11}	15.1	1.3×10^{5}	9×10^{-1}
$^{87}Rb \rightarrow {}^{87}Sr\,e^{-}\bar{\nu}_e$	4.75×10^{10}	27.83	8.8×10^{5}	8.0×10^{1}
$^{187}Re \rightarrow {}^{187}Os\,e^{-}\bar{\nu}_e$	4.15×10^{10}	62.6	1.1×10^{6}	8×10^{-4}
$^{176}Lu \rightarrow {}^{176}Hf\,e^{-}$	3.78×10^{10}	2.61	5.5×10^{4}	4×10^{-2}
$^{232}Th \rightarrow {}^{228}Ra\,\alpha$	1.405×10^{10}	100	4.05×10^{6}	3.5×10^{2}
$^{238}U \rightarrow {}^{234}Th\,\alpha$	4.468×10^{9}	99.275	1.2×10^{7}	4.7×10^{2}
$^{40}K \rightarrow {}^{40}Ca\,e^{-}\bar{\nu}_e$ 89% $\rightarrow {}^{40}Ar\,\nu_e$ 11%	1.28×10^{9}	0.0117	3.0×10^{4}	6.3×10^{2}
$^{235}U \rightarrow {}^{231}Th\,\alpha$	7.038×10^{8}	0.72	5.7×10^{5}	1.7×10^{1}
$^{146}Sm \rightarrow {}^{142}Nd\,\alpha$	1.03×10^{8}	$<10^{-7}$	<1	$<10^{-4}$

"Fundamentals In Nuclear Physics From Nuclear Structure to Cosmology" - Springer

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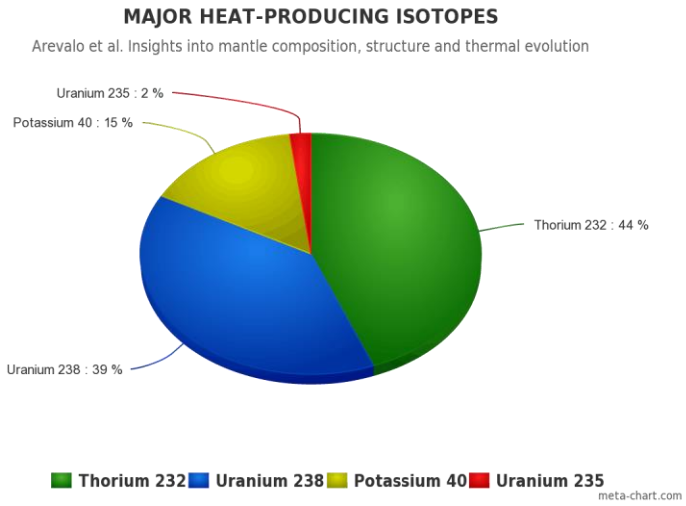
^{40}K

These four radioactive isotopes are responsible for the majority of radiogenic heat in the Earth



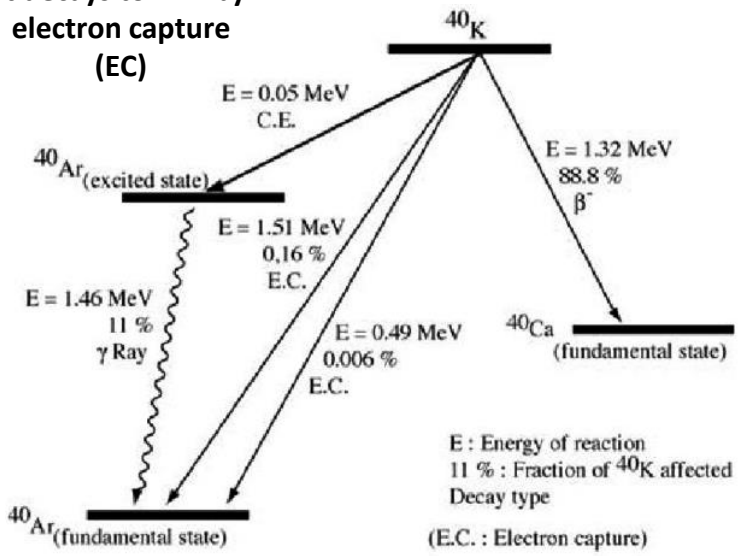
Stable 93.26% $^{39}_{19}K$

Radioactive 0.0117% $^{40}_{19}K$

Stable 6.73% $^{41}_{19}K$

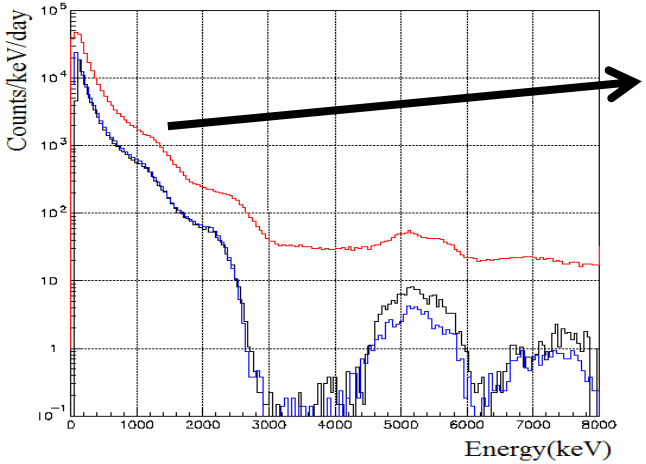
Half-life = 1.2×10^9 years

10.72% of events, it decays to ^{40}Ar by electron capture (EC)



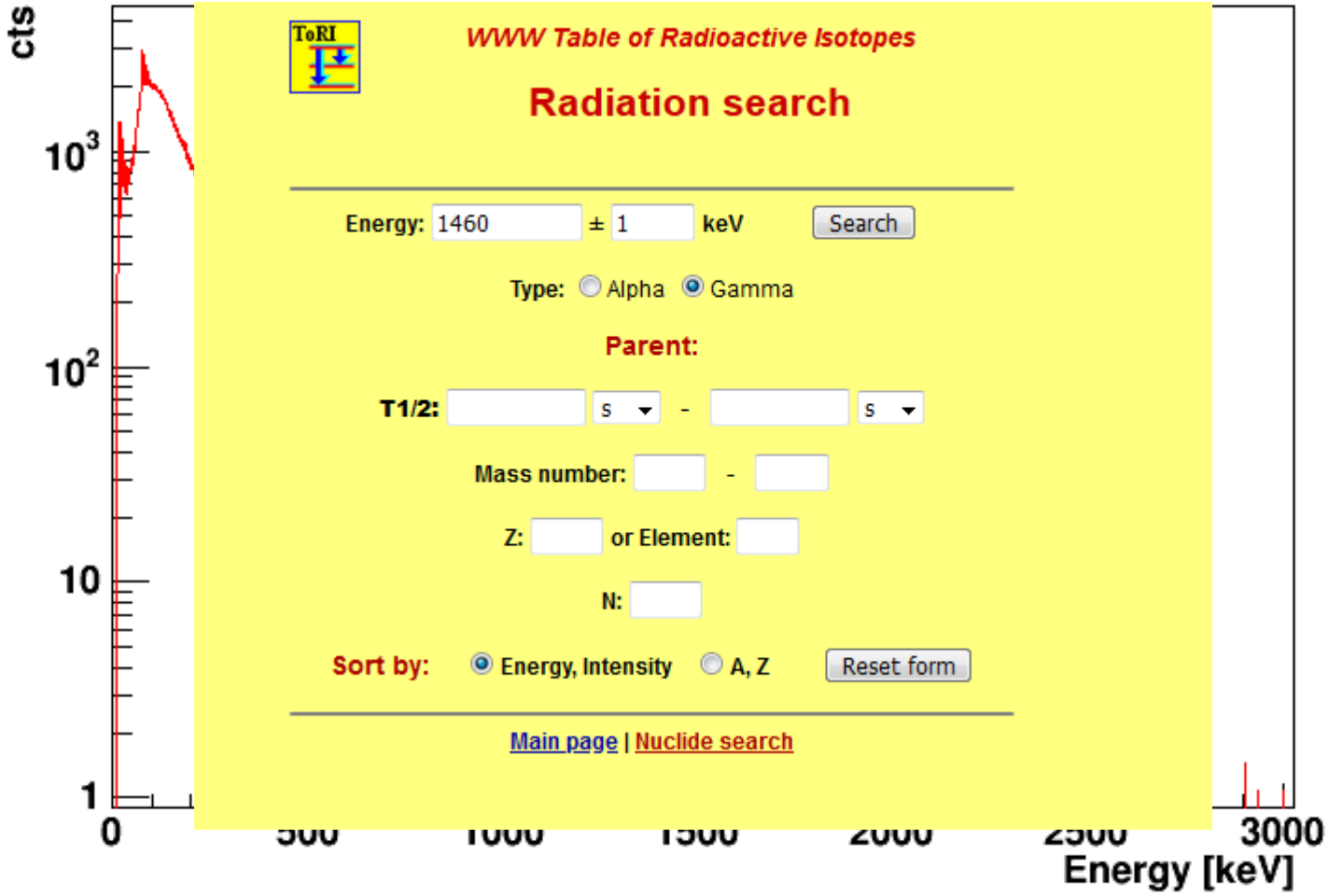
Potassium is one of the most common element on the earth crust

^{40}K emits detectable gamma rays



1.46 MeV from ^{40}K

HPGe spectrum on surface



http://nucleardata.nuclear.lu.se/toi/radSearch.asp

^{238}U, ^{235}U, ^{232}Th

They do not decay directly to a stable state: each initiate a **decay chain**
In between, there are altogether 42 radionuclides of 13 elements

The mass number of the members may be expressed as four times an appropriate integer (n) plus the constant for that series. Three natural occurring:

Thorium series (4n): ^{232}Th ($t_{1/2}$ =1.4·10^{10} y) → 10 radionuclides → ^{208}Pb
Uranium series (4n+2) : ^{238}U ($t_{1/2}$= 4.5·10^{9} y) → 17 radionuclides → ^{206}Pb
Actinium series (4n+3): ^{235}U ($t_{1/2}$= 7·10^{8} y) → 15 radionuclides → ^{207}Pb

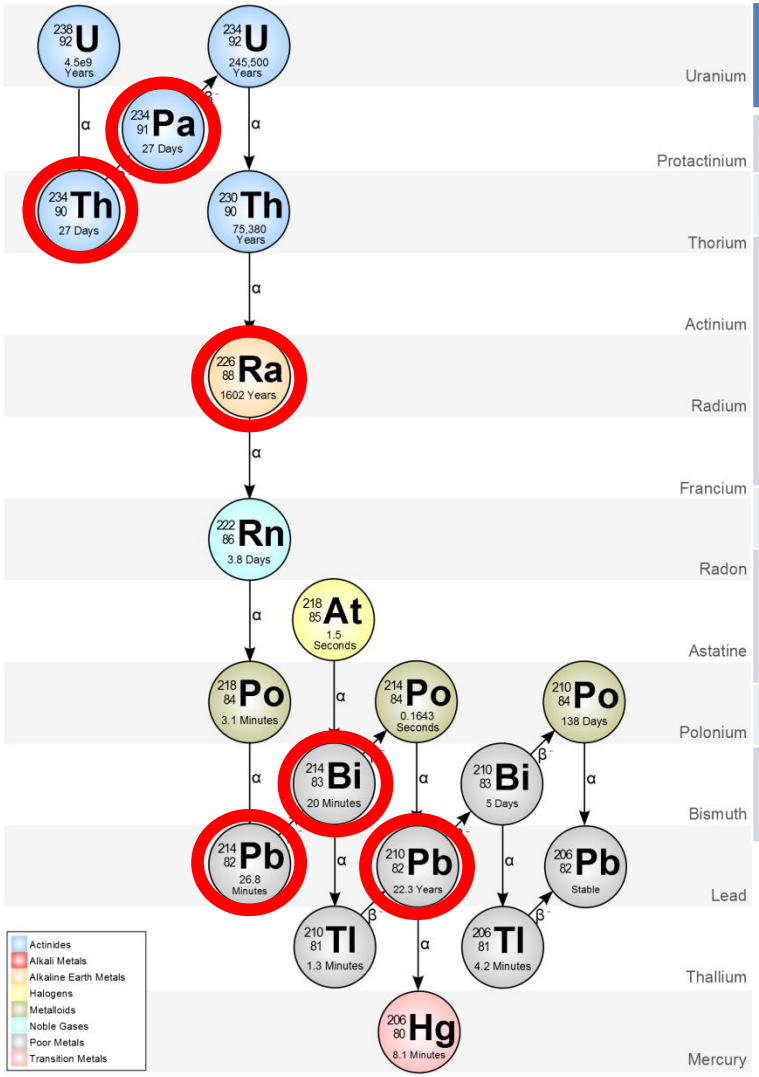
The neptunium series with Np-237 (with a half-life of 2 million years) 4n+1 series do not occur naturally, because the half-life of the longest lived isotope in the series is short compared to the age of the earth. Np decay chain has no long-lived nuclei heavier than ^{209}Bi.
The chain thus consists of a single decay $^{209}Bi \rightarrow {}^{205}Tl_{(stable)}$ ($t_{1/2}$ = 1.9 × 10^{19} y)

In these chains they are alpha and beta emitters, most of which do not emit detectable gamma rays:

- Gamma spectroscopy alone is not sufficient

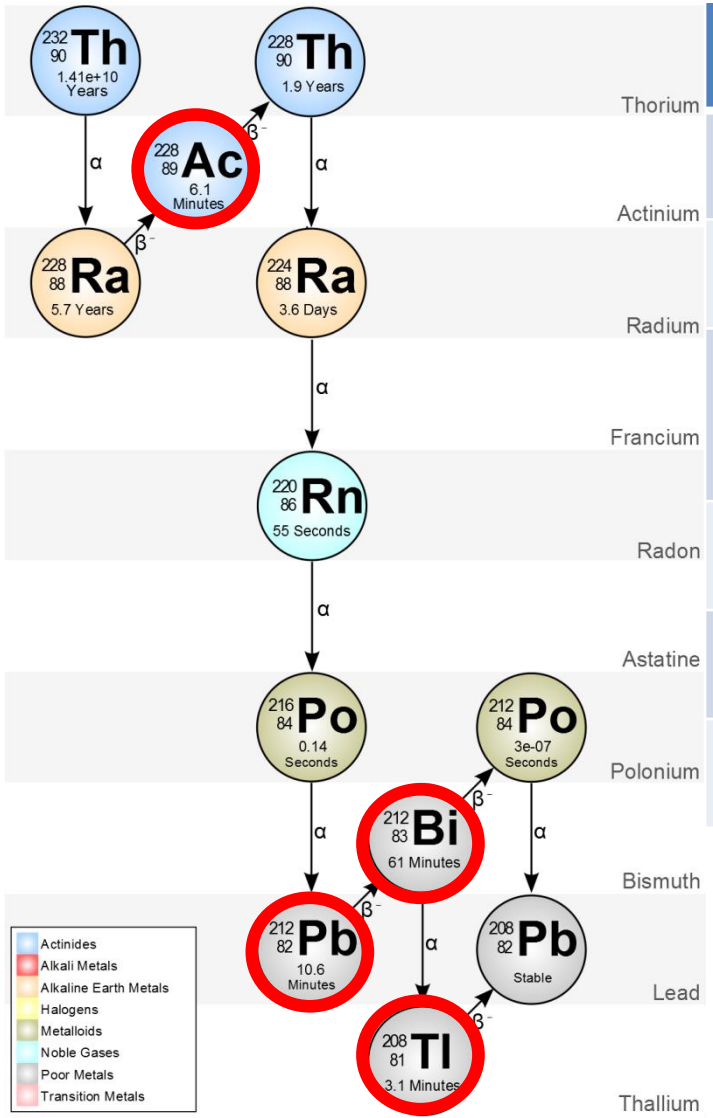
(the determination of the activity requires radiochemical separations)

^{238}U chain



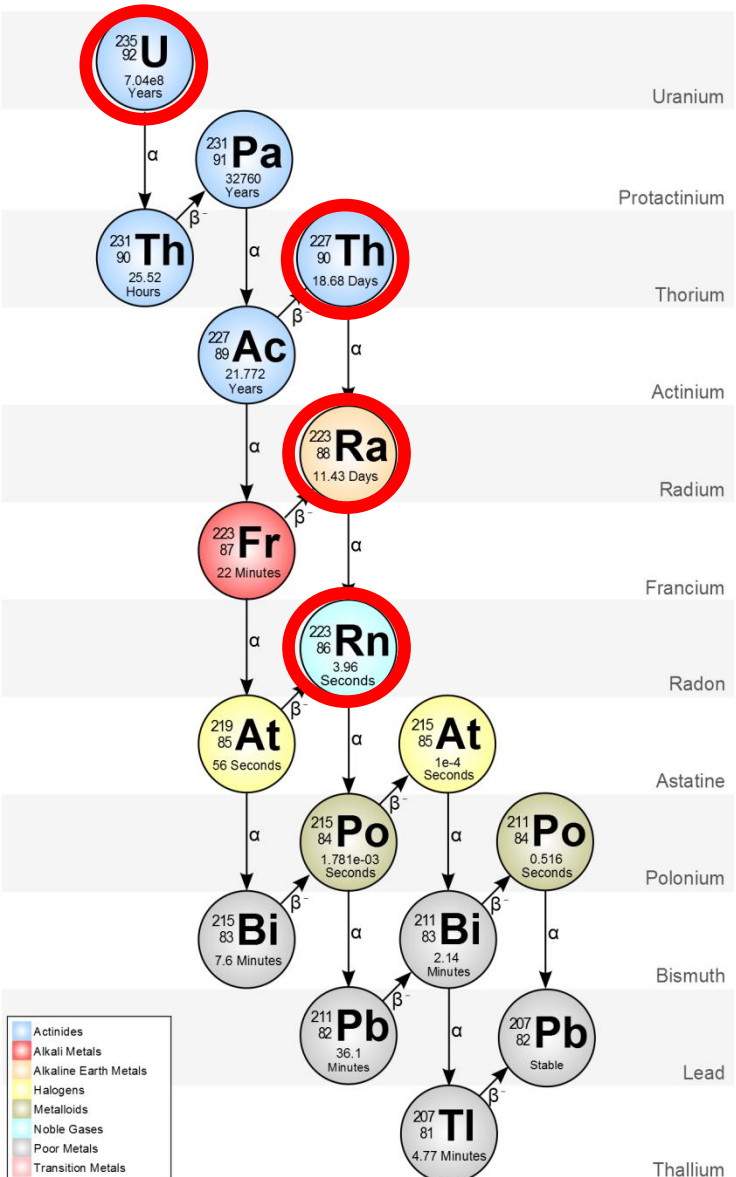
Nuclide	Half-Life	Energy [keV]	Emission Prob.	Comments
Th-234	24.10 d	63.28	4.8(6)	-
Pa-234m	1.17 m	1001.03	1.021(15)	-
Ra-226	5.862E5 d	186.21	3.555(19)	57.1% composite peak – Ra-235 and Th-230 interference correction needed*
Pb-214	26.8 m	351.93	35.60(7)	-
		295.22	18.414(36)	Insignificant interference from Bi-212
Bi-214	19.9 m	1764.49	15.31(5)	-
Pb-210	8.14E3 d	46.54	4.25(5)	Slight interference from Pa-231

^{232}Th chain



Nuclide	Half-Life	Energy [keV]	Emission Prob.	Comments
Ac-228	6.15 h	911.20	25.8(4)	Subject to TCS
		968.97	15.8	Subject to TCS
Pb-212	10.64h	238.63	43.6(3)	Deconvolution with 242 keV of Pb-214 needed
Bi-212	60.54 m	1620.74	1.51(3)	-
Tl-208*	3.06 m	583.19	85(5)	Subject to TCS**
		860.56	12.5(2)	Subject to TCS**

^{235}U chain



Nuclide	Half-Life	Energy [keV]	Emission Prob.	Comments
U-235	2.571E11 d	185.72	57.2(8)	44.8% of composite peak – Ra-223. Ra-226 and Th-230 correction needed
		143.7	10.96(8)	Th-230 interference correction needed
		163.33	5.08(7)	-
Th-227	18.718 d	235.96	12.6(6)	-
		256.23	6.8(4)	-
Ra-223	11.43 d	269.46	13.7(4)	Interference from Ac-228
Rn-219	3.96 s	271.23	10.8(7)	Interference form Ac-228 and Ra-223
		401.81	6.4(5)	-

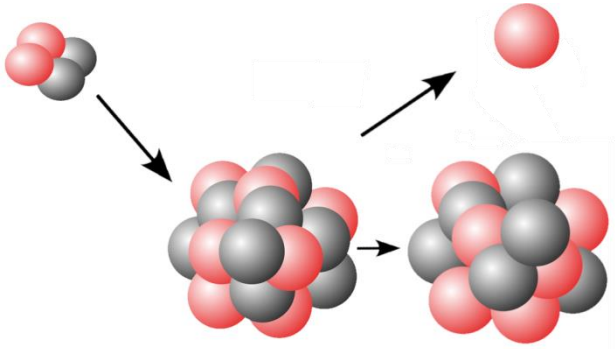
uranium series	actinium series	thorium series
$^{238}U_{92}$ 4.468 Gyr		
$^{234}Th_{90}$ 24.10 d		
$^{234}Pa_{91}$ 1.17 m		
$^{234}U_{92}$ 245.5 kyr	$^{235}U_{92}$ 0.7038 Gyr	
$^{230}Th_{90}$ 75.38 kyr	$^{231}Th_{90}$ 25.52 h	$^{232}Th_{90}$ 14.05 Gyr
$^{226}Ra_{88}$ 1600 y	$^{231}Pa_{91}$ 32760 y	$^{228}Ra_{88}$ 5.75 y
$^{222}Rn_{86}$ 3.8235 d	$^{227}Ac_{89}$ 21.773 y	$^{228}Ac_{89}$ 6.15 h
$^{218}Po_{84}$ 3.10 m	$^{227}Th_{90}$ 18.72 d	$^{228}Th_{90}$ 1.9116 y
$^{214}Pb_{82}$ 26.8 m	$^{223}Ra_{88}$ 11.435 d	$^{224}Ra_{88}$ 3.66 d
$^{214}Bi_{83}$ 19.9 m	$^{219}Rn_{86}$ 3.96 s	$^{220}Rn_{86}$ 55.6 s
$^{214}Po_{84}$ 164.3 µs	$^{215}Po_{84}$ 1.781 ms	$^{216}Po_{84}$ 0.145 s
$^{210}Pb_{82}$ 22.3 y	$^{211}Pb_{82}$ 36.1 m	$^{212}Pb_{82}$ 10.64 h
$^{210}Bi_{83}$ 5.013 d	$^{211}Bi_{83}$ 2.14 m	$^{212}Bi_{83}$ 60.55 m
$^{210}Po_{84}$ 138.376 d	$^{207}Tl_{81}$ 4.77 m	64% $^{212}Po_{84}$ 0.299µs
		36% $^{208}Tl_{81}$ 3.053 m
$^{206}Pb_{82} > 10^{20}$ yr	$^{207}Pb_{82} > 10^{20}$ yr	$^{208}Pb_{82} > 10^{20}$ yr

Important alpha emitters

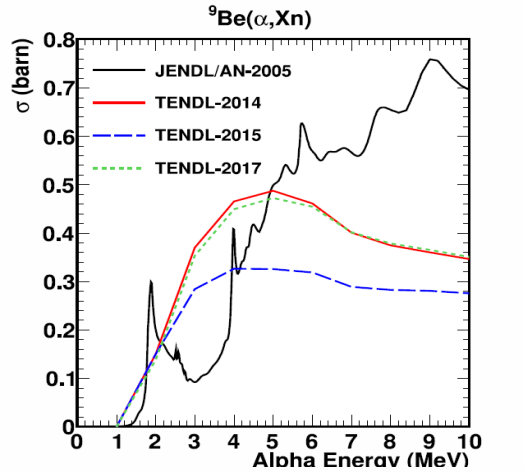
- Th-232: ^{216}Po (6.8 MeV) and ^{212}Po (8.8 MeV),
- U-238: ^{218}Po (6.0 MeV), ^{214}Po (7.7 MeV), and ^{210}Po (5.3 MeV).

Important gamma emitters: Tl, Bi…

(α,n) reactions

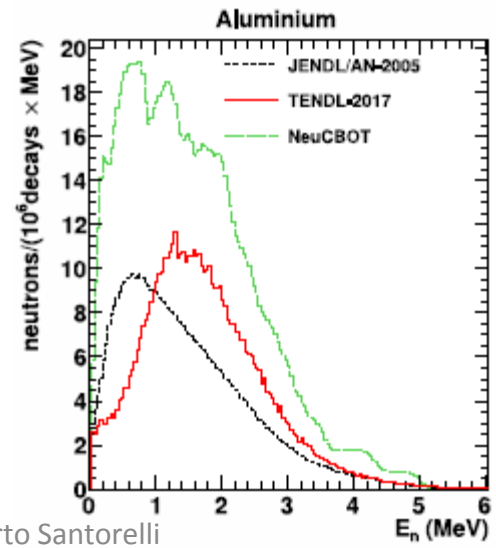


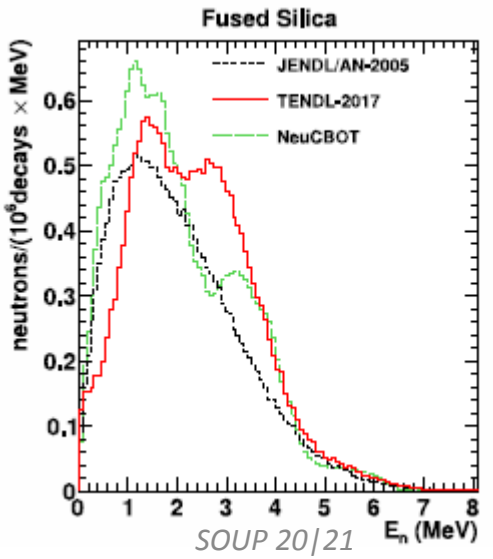
$$Y_i(E_\alpha) = \frac{\eta_i}{\eta} \int_0^{E_\alpha} \frac{\sigma^i_{(\alpha,Xn)}(E)}{\varepsilon(E)} dE$$

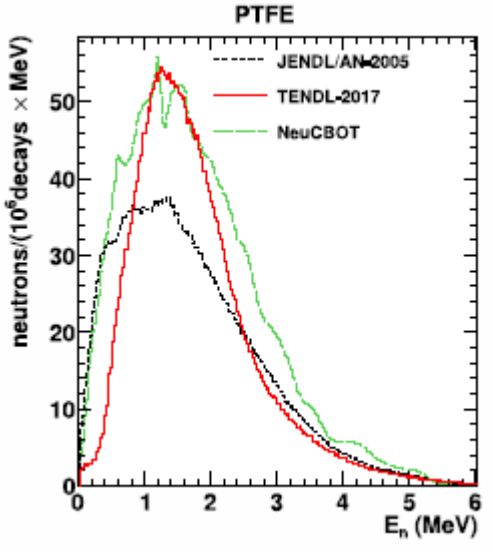


E_α is the initial energy of the α particle;
η_i is the number density of nuclide i;
η is the number density of the material;
$\sigma^i(\alpha,Xn)(E)$ is the neutron production x-sec for the nuclide i
$\varepsilon(E) = -\frac{dE}{dx}$ is the stopping power of the material

http://win.ciemat.es/SaG4n/







The primordial alpha emitters produce secondary neutrons mainly through (α, n) reactions from the alpha decays in the uranium and thorium chains.

Formaggio & Martoff

Type of rock	U (ppm)	Th (ppm)	U(α, n)	Th(α, n)	Fission	Total yield
	Concentration (ppm)		(neutrons/g/y)			
Granite	5	11	7.85	7.755	2.33	17.9
Limestone	1	1	0.64	0.285	0.467	1.4
Sandstone	1	1	0.837	0.38	0.467	1.7
Granite A	1.32	7.79	2.24	5.92	0.62	8.8
Granite B	6.25	4.59	10.62	3.49	2.92	17.0
Granite C	1.83	4.38	3.11	3.33	0.85	7.3
Salt I	0.30	2.06	1.60	4.77	0.14	6.5
Salt II	0.13	1.80	4.17	0.69	0.06	4.9

U and Th concentrations for various types of rocks, along with estimated neutron production

Equilibrium in the chain

Equilibrium means that the **activities** of the parent and daughter nuclides are the same

- (Transient) Equilibrium: the half-life of the parent nuclide is longer than that of the daughter
- No-equilibrium: the half-life of the parent nuclide is shorter than that of the daughter

If no progeny atoms are present at time $t = 0$, the number N_2 of progeny atoms at any later time t is:

$$N_2(t) = [\lambda_1/(\lambda_2 - \lambda_1)]N_0(e^{-\lambda_1 t})$$ Bateman equation

N_0 is the number of parent atoms present at time $N_1(t = 0)$
λ_1 is the decay constant of the parent,
λ_2 is the decay constant of the progeny

If $(N_2)_0$ progeny atoms are present at time $t = 0$, the expression for $N2$ is written

$$N_2(t) = (N_2)_0 e^{-\lambda_2 t} + [\lambda_1/(\lambda_2 - \lambda_1)]N_0(e^{-\lambda_1 t} - e^{-\lambda_2 t})$$

The apparent half-life of the progeny reflects the simultaneous production and decay of the progeny. The ratio of activities $A1$ and $A2$ for X and Y, respectively, is

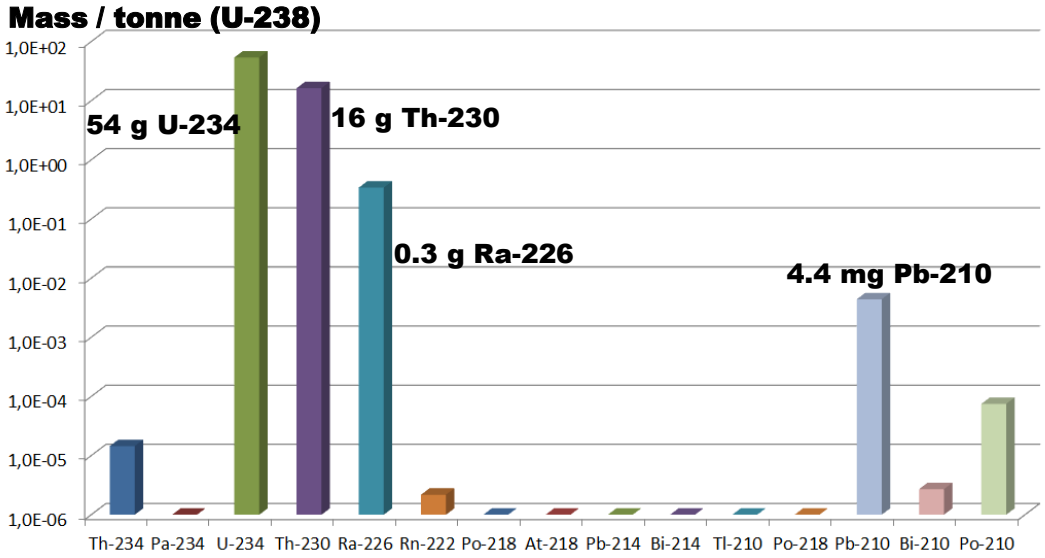
$$A_1/A_2 = (\lambda_2 - \lambda_1)/\lambda_2$$

Secular equilibrium

"Secular equilibrium": the half-life of the parent nuclide is very long and the half-life of the daughter nuclide is considerably shorter than that of the parent

A radioactive nuclide is decaying at the same rate at which it is being produced:
→ the number of atoms present remains constant (over a certain amount of time)

Over the 4.5 billion years of the Earth's history, all three of these decay chains have reached equilibria between the parent nucleus and the various descendants (supposing that the system has been closed millions of years)



Minerals that contain uranium and thorium also contain their daughters (virtually all species in the chain)

Disequilibrium in natural decay chains

Equilibrium in the decay series ***is rarely achieved*** in most surface and near-surface geological environments, as a result of physical or chemical processes:

- chemical, artificial or natural processes (e.g. dissolution into groundwater) can remove one or more components of the chain.

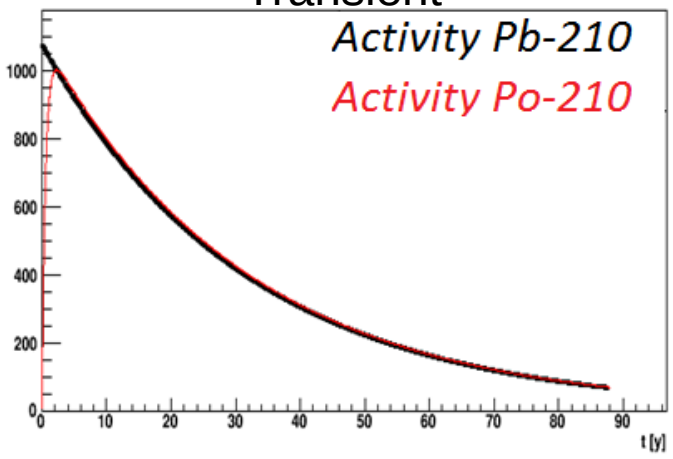
If for example uranium is dissolved from a primary uranium-bearing mineral by oxidation the remaining radionuclides in the ^{238}U series will be supported by its most long-lived radionuclide which is ^{230}Th. If the dissolved uranium will then be precipitated somewhere out of the system a new equilibrium will start to develop.

The time required to attain the equilibrium is governed by the most long-lived daughter radionuclide in the series, ^{230}Th in case of ^{238}U series. The half-life of ^{230}Th is 75000 years and this time is required to attain 50% of the equilibrium.

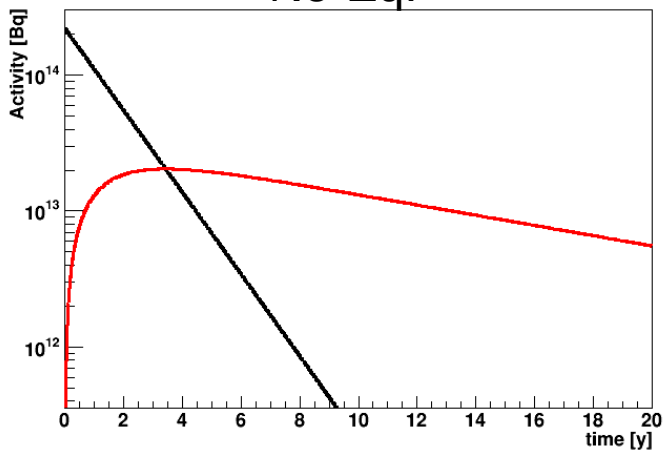
The disequilibria can be utilized in dating geological events.

Examples

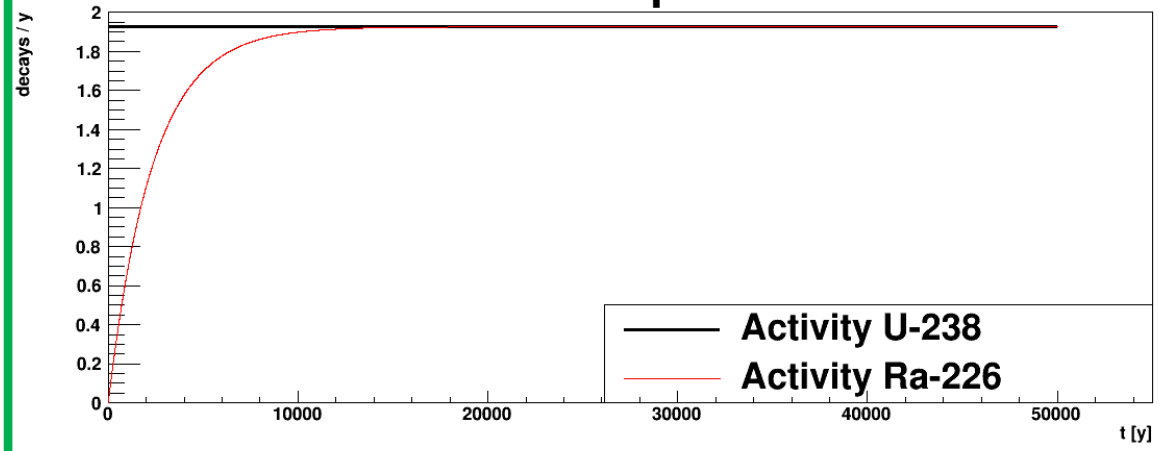
Transient

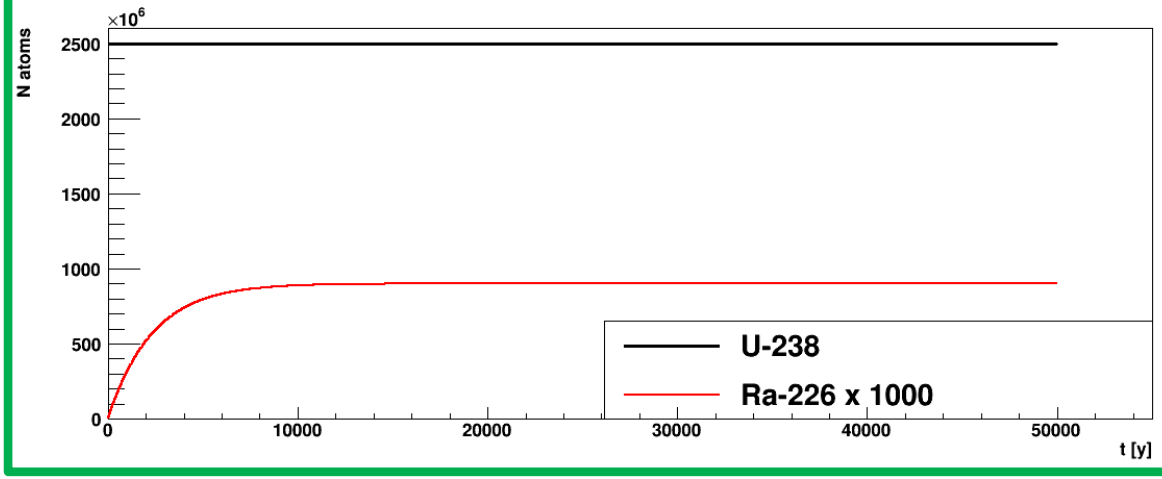


No-Eq.



Secular equilibrium

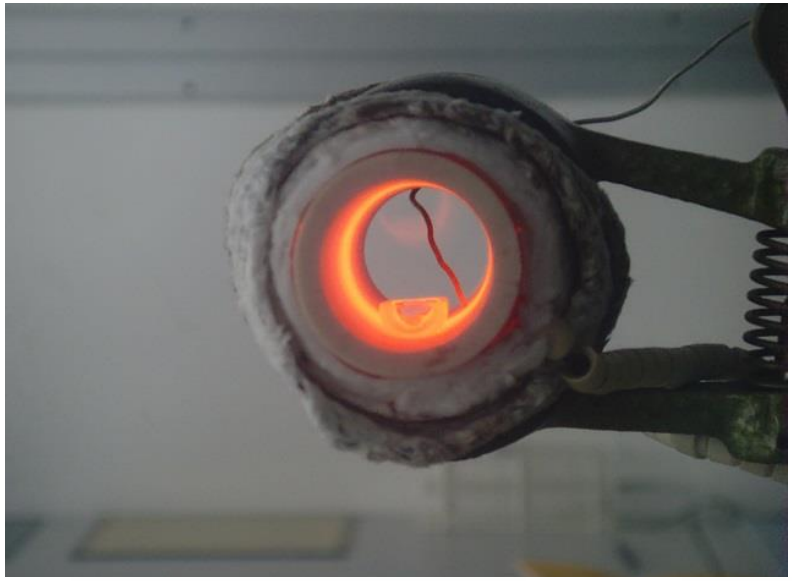




Example of equilibrium broken: Radon

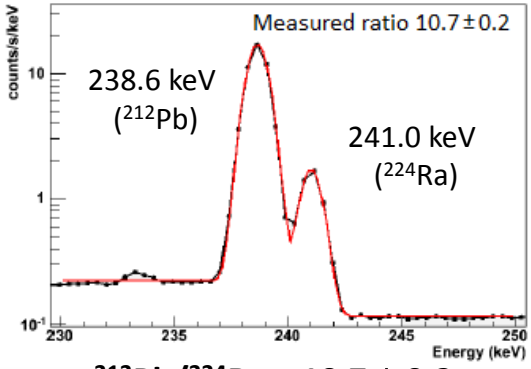
Equilibrium broken due to Rn gas emanation during the procedure

$$^{228}Th \rightarrow {}^{224}Ra \rightarrow {}^{220}Rn \rightarrow {}^{216}Po \rightarrow {}^{212}Pb \rightarrow {}^{212}Bi \left| \begin{array}{l} \rightarrow {}^{212}Po \\ \\ \rightarrow {}^{208}Tl \end{array} \right| \rightarrow {}^{208}Pb$$



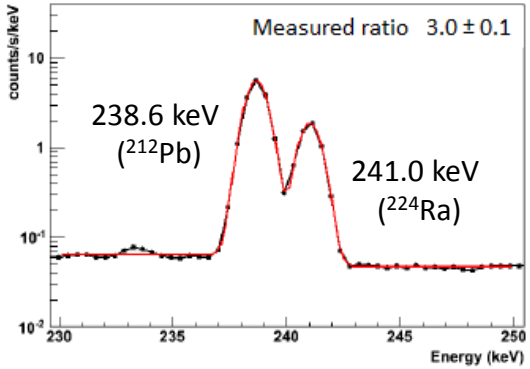
Relative peak height ratio:
$^{212}Pb/^{224}Ra \cong 10.6$

Before the treatment



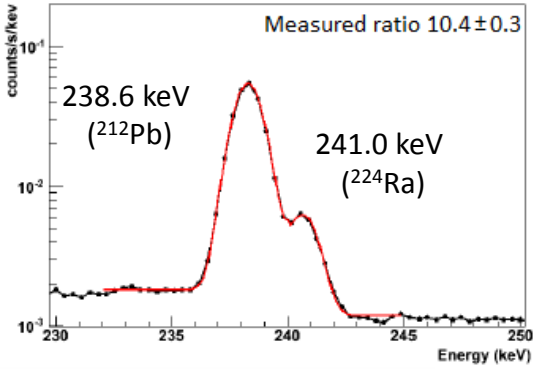
$^{212}Pb/^{224}Ra = 10.7 \pm 0.2$

1h after the treatment



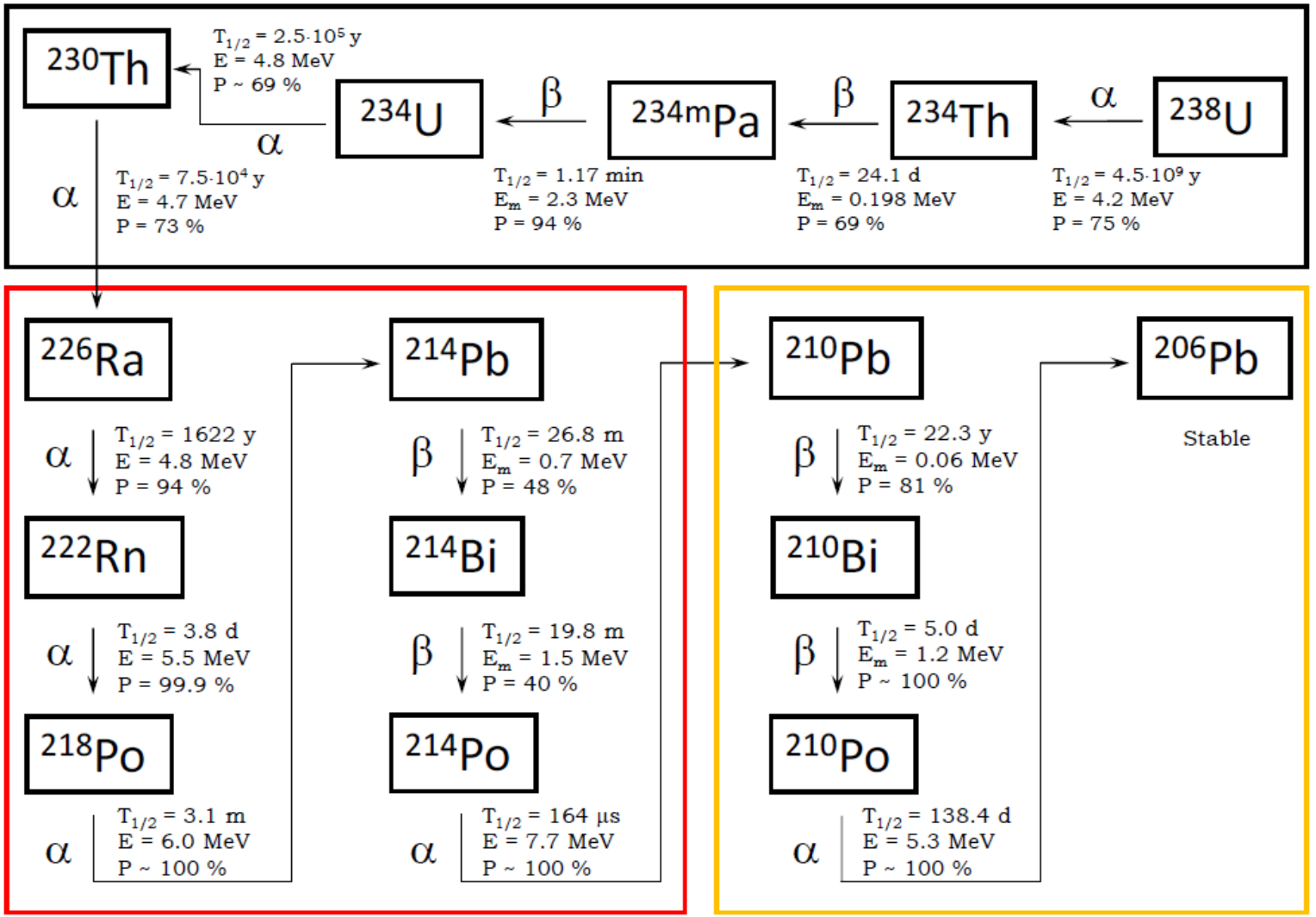
$^{212}Pb/^{224}Ra = 3.0 \pm 0.1$

2 months after

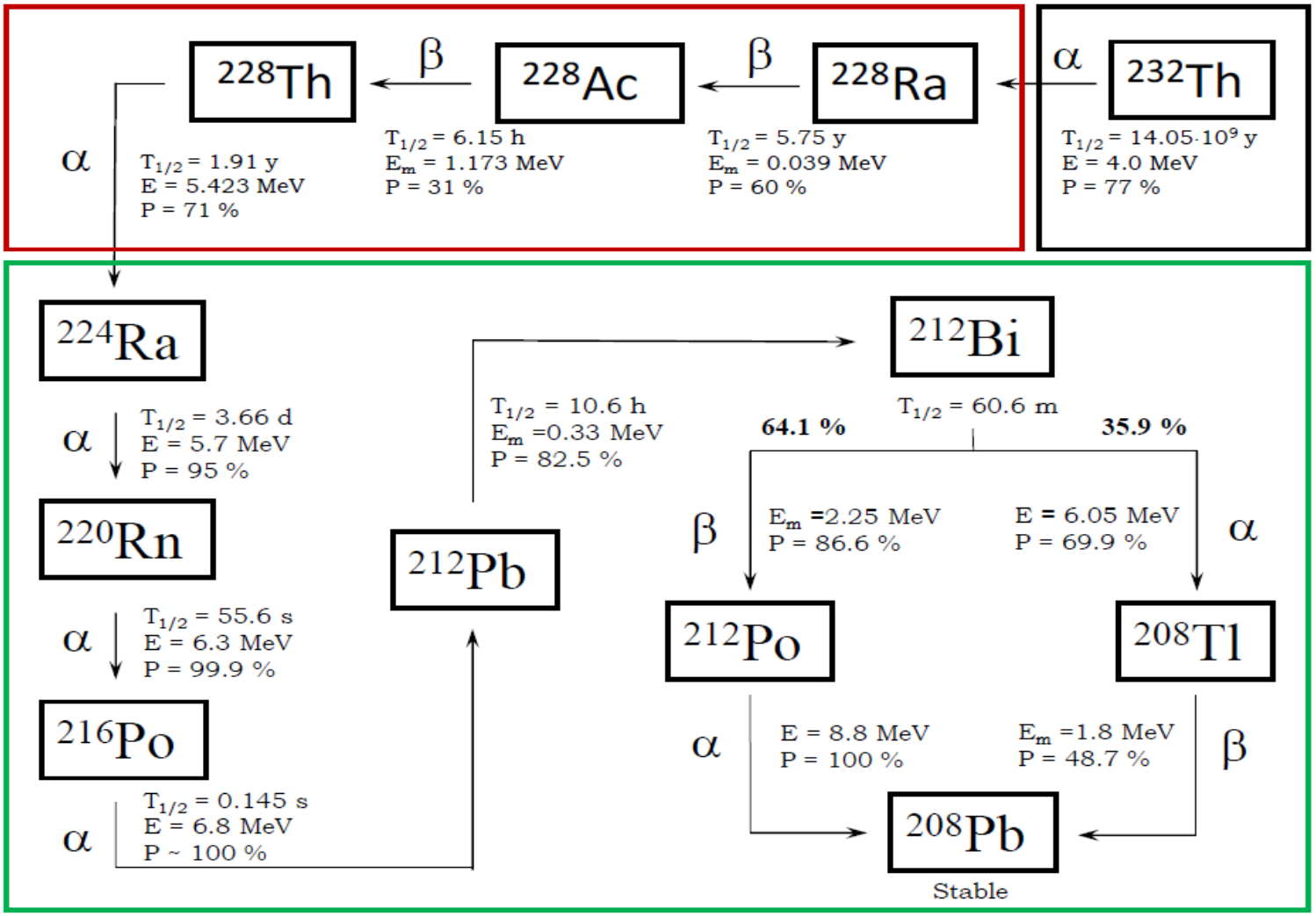


$^{212}Pb/^{224}Ra = 10.4 \pm 0.3$

^{238}U chain


238U
α
T1/2 = 4.5·10^9 y
E = 4.2 MeV
P = 75 %
234Th
β
T1/2 = 24.1 d
Em = 0.198 MeV
P = 69 %
234mPa
β
T1/2 = 1.17 min
Em = 2.3 MeV
P = 94 %
234U
α
T1/2 = 2.5·10^5 y
E = 4.8 MeV
P ~ 69 %
230Th
α
T1/2 = 7.5·10^4 y
E = 4.7 MeV
P = 73 %
226Ra
α
T1/2 = 1622 y
E = 4.8 MeV
P = 94 %
222Rn
α
T1/2 = 3.8 d
E = 5.5 MeV
P = 99.9 %
218Po
α
T1/2 = 3.1 m
E = 6.0 MeV
P ~ 100 %
214Pb
β
T1/2 = 26.8 m
Em = 0.7 MeV
P = 48 %
214Bi
β
T1/2 = 19.8 m
Em = 1.5 MeV
P = 40 %
214Po
α
T1/2 = 164 μs
E = 7.7 MeV
P ~ 100 %
210Pb
β
T1/2 = 22.3 y
Em = 0.06 MeV
P = 81 %
210Bi
β
T1/2 = 5.0 d
Em = 1.2 MeV
P ~ 100 %
210Po
α
T1/2 = 138.4 d
E = 5.3 MeV
P ~ 100 %
206Pb
Stable

^{232}Th chain


232Th
α
T1/2 = 14.05·10^9 y
E = 4.0 MeV
P = 77 %
228Ra
β
T1/2 = 5.75 y
Em = 0.039 MeV
P = 60 %
228Ac
β
T1/2 = 6.15 h
Em = 1.173 MeV
P = 31 %
228Th
α
T1/2 = 1.91 y
E = 5.423 MeV
P = 71 %
224Ra
α
T1/2 = 3.66 d
E = 5.7 MeV
P = 95 %
220Rn
α
T1/2 = 55.6 s
E = 6.3 MeV
P = 99.9 %
216Po
α
T1/2 = 0.145 s
E = 6.8 MeV
P ~ 100 %
212Pb
T1/2 = 10.6 h
Em =0.33 MeV
P = 82.5 %
212Bi
T1/2 = 60.6 m
64.1 %
35.9 %
β
Em =2.25 MeV
P = 86.6 %
α
E = 6.05 MeV
P = 69.9 %
212Po
208Tl
α
E = 8.8 MeV
P = 100 %
β
Em =1.8 MeV
P = 48.7 %
208Pb
Stable

Cosmogenic radionuclides

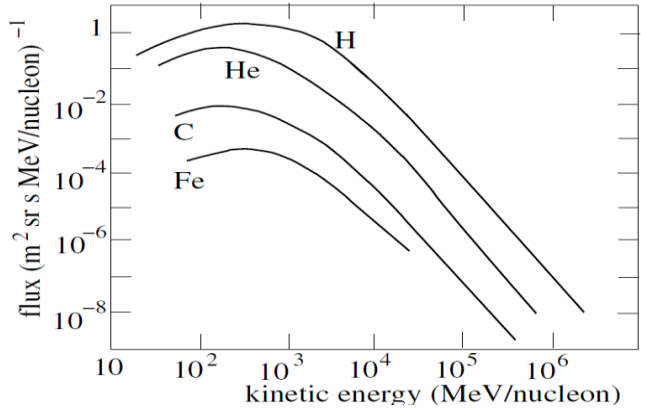
Cosmic-rays produce radioactive nuclei via their interactions with nuclei in the atmosphere and in the Earth's crust.

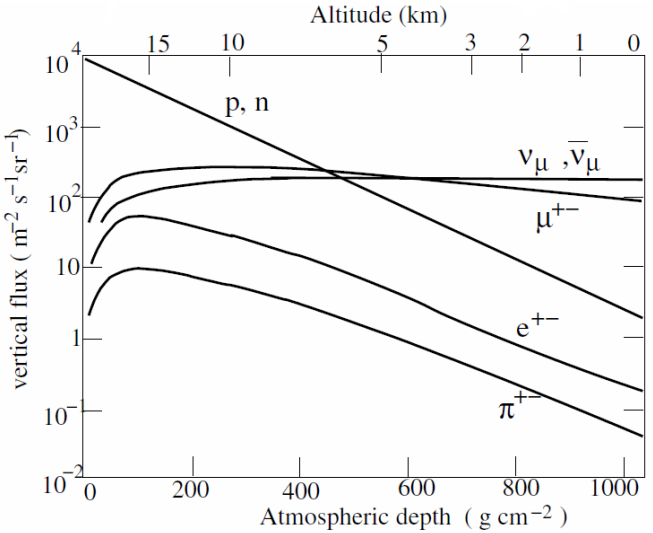
These radionuclides are isotopes of lighter elements, and their half-lives vary greatly.

Most of the cosmogenic radionuclides are attached to aerosol particles and are deposited on the ground. Some, however, are gaseous, such as ^{39}Ar (a noble gas), and thus stay in the atmosphere.

Since the intensity of cosmic radiation is in the long-term constant, the production of the cosmogenic radionuclides is rather constant. There is, however, great variation at different heights of the atmosphere and at different latitudes.

$$^{14}N + n \rightarrow {}^{14}C + p$$
$$^{14}N + n \rightarrow {}^{12}C + {}^{3}H$$





Nuclide	Half-life	Nuclide	Half-life
^{3}H	12.3 a	^{7}Be	53 d
^{10}Be	$2.5 \cdot 10^6$ a	^{14}C	5730 a
^{22}Na	2.62 a	^{26}Al	$7.4 \cdot 10^5$ a
^{32}Si	710 a	^{32}P	14.3 d
^{33}P	24.4 d	^{35}S	88 d
^{36}Cl	$3.1 \cdot 10^5$ a	^{39}Ar	269 a

Cosmogenic radionuclides

During **transport in air**, the **activation** by the hadronic component can reach specific radioactivity levels higher than the residual contamination from primordial nuclides.

High energy cosmic rays also produce radioactive nuclei through "**spallation**" reactions where one or more nucleons are removed from a nucleus
For example a neutron with kinetic energy greater than ~20 MeV can remove two nucleons for a germanium nucleus,
e.g. $n + {}^{70}Ge \rightarrow 3n + {}^{68}Ge$ $\quad t_{1/2}({}^{68}Ge) = 270.7\,day$

This reaction results in a radioactivity of ~0.3 mBq kg^{-1} in germanium crystals produced at the Earth's surface.
This is the most important source of intrinsic radioactivity in the HPGe crystals.

Materials have to be **stored underground** (the production the cosmogenic radionuclides stops and the activity decays away).

Materials extracted UG should be largely depleted in cosmogenic isotopes.

Rates of production of long-lived radioisotopes at sea level (atoms/kg/day)

Copper

	^{46}Sc	^{48}V	^{54}Mn	^{56}Co	^{57}Co	^{58}Co	^{59}Fe	^{60}Co
Half-life[27,108]	83.787(16)	15.9735	312.19(3)	77.236	271.81(4)	70.85(3)	44.494	5.2711(8)
units	d	d	d	d	d	d	d	y
Measurement [202]	2.18 ± 0.74	4.5 ± 1.6	8.85 ± 0.86	9.5 ± 1.2	74 ± 17	67.9 ± 3.7	18.7 ± 4.9	86.4 ± 7.8
Measurement [184]	$2.33^{+0.95}_{-0.78}$	$3.4^{+1.6}_{-1.3}$	$13.3^{+3.0}_{-2.9}$	$9.3^{+1.2}_{-1.4}$	$44.8^{+8.6}_{-8.2}$	$68.9^{+5.4}_{-5.0}$	$4.1^{+1.4}_{-1.2}$	$29.4^{+7.1}_{-5.9}$
ACTIVIA (MENDL-2P) [36]	3.1		12.4	14.1	36.4	38.1	1.8	9.7
ACTIVIA [36,184]	3.1		14.3	8.7	32.5	56.6	4.2	26.3
COSMO [184]	1.5	3.1	13.5	7.0	30.2	54.6	4.3	25.7
ACTIVIA [46]	4.1		30.0	20.1	77.5	138.1	10.5	66.1
ACTIVIA [99]	3		16	9	34	60	2	29
GEANT4 [46]	1.2		12.3	10.3	67.2	57.3	8.8	64.6
TALYS [94]			16.2		56.2			46.4
MENDL+YIELDX [43]	2.7		27.7	20.0	74.1	123.0	4.9	55.4
CONUS [99]	3		14	10	50	76	5	92

SS

Isotope	^{7}Be	^{46}Sc	^{48}V	^{54}Mn	^{56}Co	^{58}Co
Half-life (d) [27,108]	53.22(6)	83.787(16)	15.9735	312.19(3)	77.236	70.85(3)
Measurement [202]	389 ± 60	19.0 ± 3.5	34.6 ± 3.5	233 ± 26	20.7 ± 3.5	51.8 ± 7.8
GEANT4 [46]	0.05	8.8		230	16	90
ACTIVIA [46]	2.05	18		191	131	13

Lead

	^{194}Hg	^{202}Pb	^{207}Bi
Half-life (y) [27,108]	444	$5.25\ 10^{4}$	32.9
Measurement [205]	8.0 ± 1.3	120 ± 25	<0.17
TALYS [205]	16	77	

(Universe 6 (2020) 10, 162)

Artificial radionuclides

Sources of artificial radionuclides:

- *nuclear weapons production and explosions;*
- *nuclear energy production;*
- *radionuclide production by reactors and accelerators (medical application etc…)*

Artificial radionuclides form the **largest group of radionuclides**, comprising more than two thousand nuclides.

The explosion clouds of the most powerful tests entered the upper part of the atmosphere, the stratosphere, from where the radioactive pollutants have deposited on a global scale.

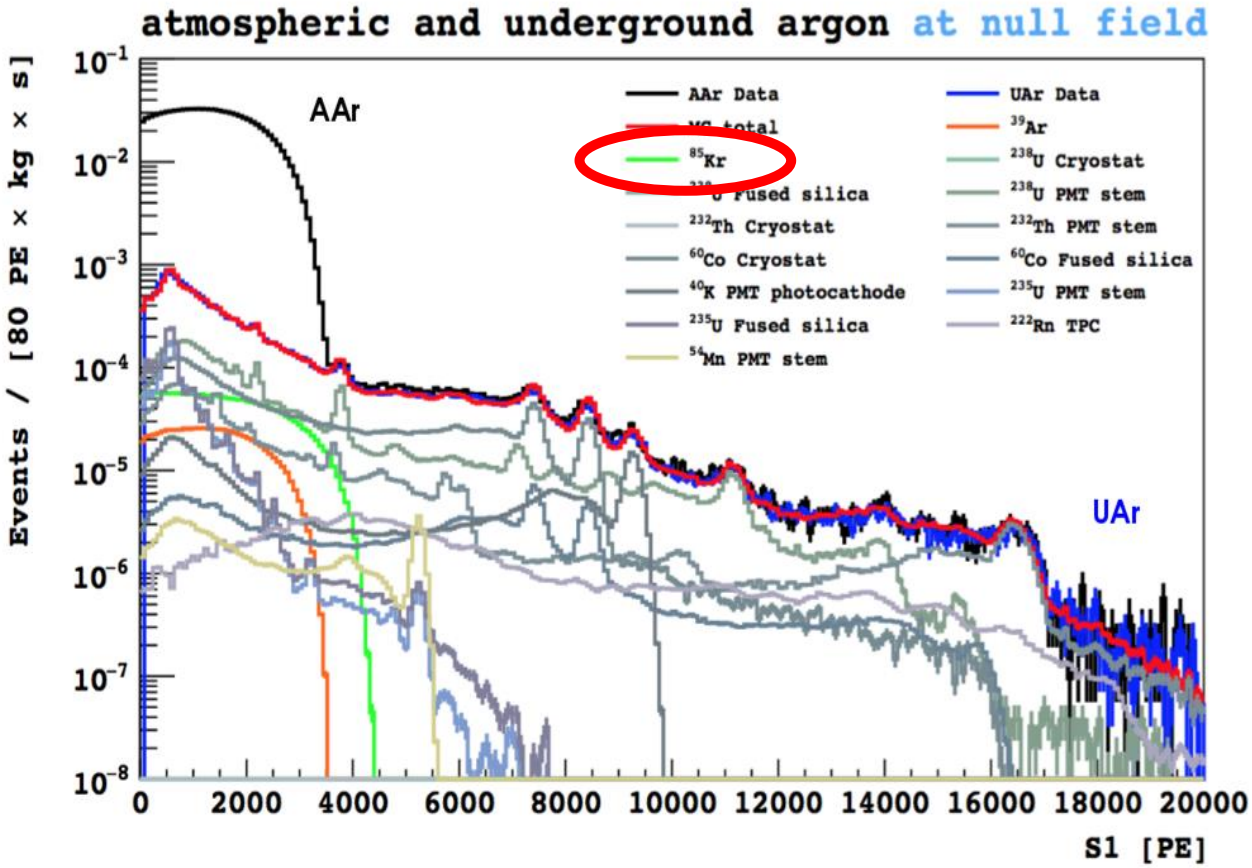
Cs-137 ($T_{1/2}$=30.2), along with iodine-131, xenon-133, and strontium-90, has been released into the environment by almost all nuclear weapons tests and major nuclear accidents.

The mean Cs-137 contamination in East Europe following the Chernobyl disaster was several kBq/m^2.

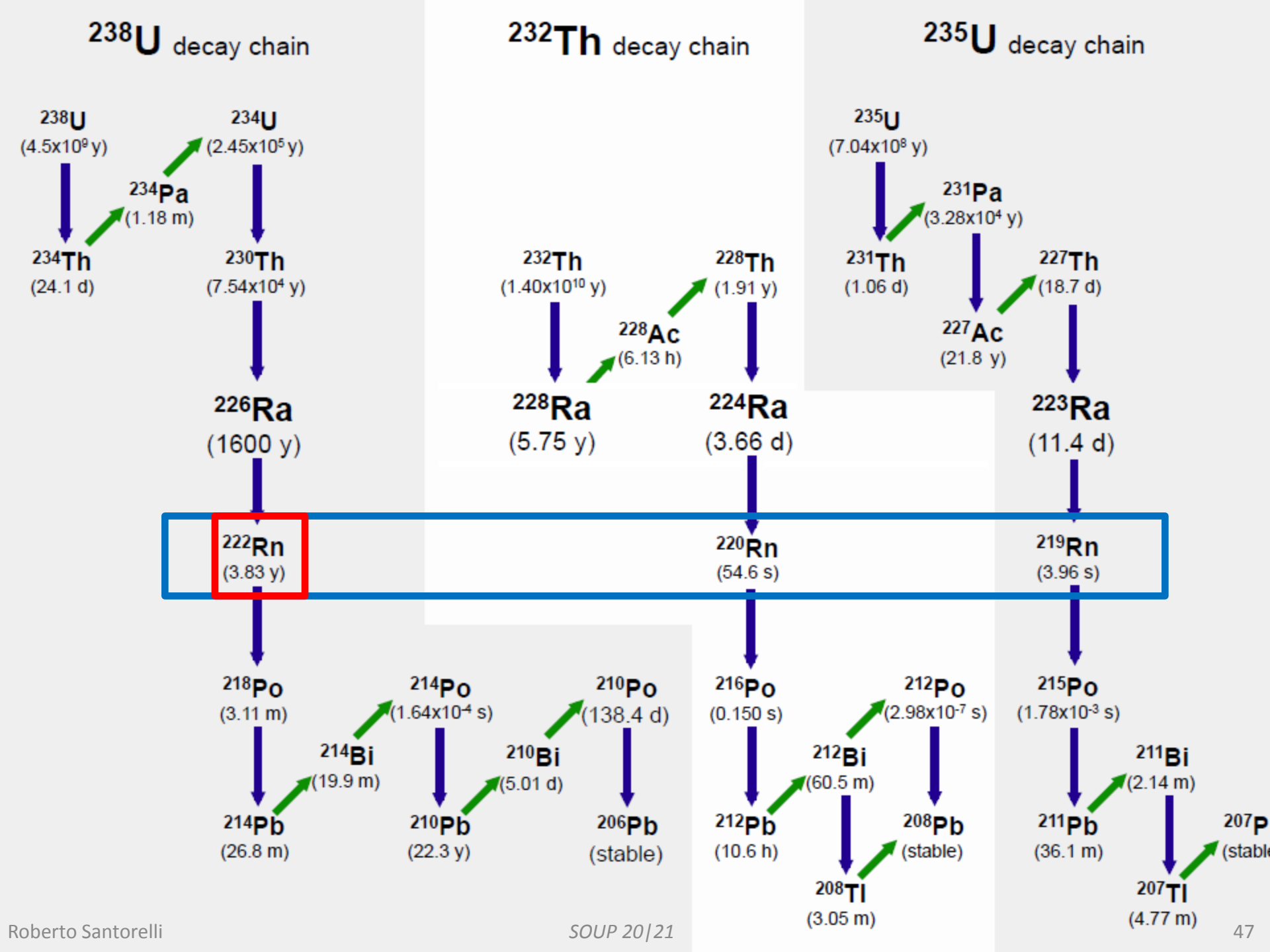
Artificial radionuclides

Kr-85 ($T_{1/2}$=10 y) is one of the seven common medium-lived fission products

kr-85 is produced in small quantities by the interaction of cosmic rays with stable Kr-84 in the atmosphere. Natural sources maintain an equilibrium inventory of about 0.09 PBq in the atmosphere. The Fukushima accident released an estimated 44–84 PBq




DANGER
RADON GAS


238U decay chain
232Th decay chain
235U decay chain
238U (4.5x10⁹ y)
234U (2.45x10⁵ y)
234Pa (1.18 m)
234Th (24.1 d)
230Th (7.54x10⁴ y)
226Ra (1600 y)
222Rn (3.83 y)
218Po (3.11 m)
214Po (1.64x10⁻⁴ s)
214Bi (19.9 m)
210Po (138.4 d)
210Bi (5.01 d)
214Pb (26.8 m)
210Pb (22.3 y)
206Pb (stable)
232Th (1.40x10¹⁰ y)
228Th (1.91 y)
228Ac (6.13 h)
228Ra (5.75 y)
224Ra (3.66 d)
220Rn (54.6 s)
216Po (0.150 s)
212Po (2.98x10⁻⁷ s)
212Bi (60.5 m)
212Pb (10.6 h)
208Pb (stable)
208Tl (3.05 m)
235U (7.04x10⁸ y)
231Pa (3.28x10⁴ y)
231Th (1.06 d)
227Th (18.7 d)
227Ac (21.8 y)
223Ra (11.4 d)
219Rn (3.96 s)
215Po (1.78x10⁻³ s)
211Bi (2.14 m)
211Pb (36.1 m)
207Tl (4.77 m)

$^{(222)}$Rn problem

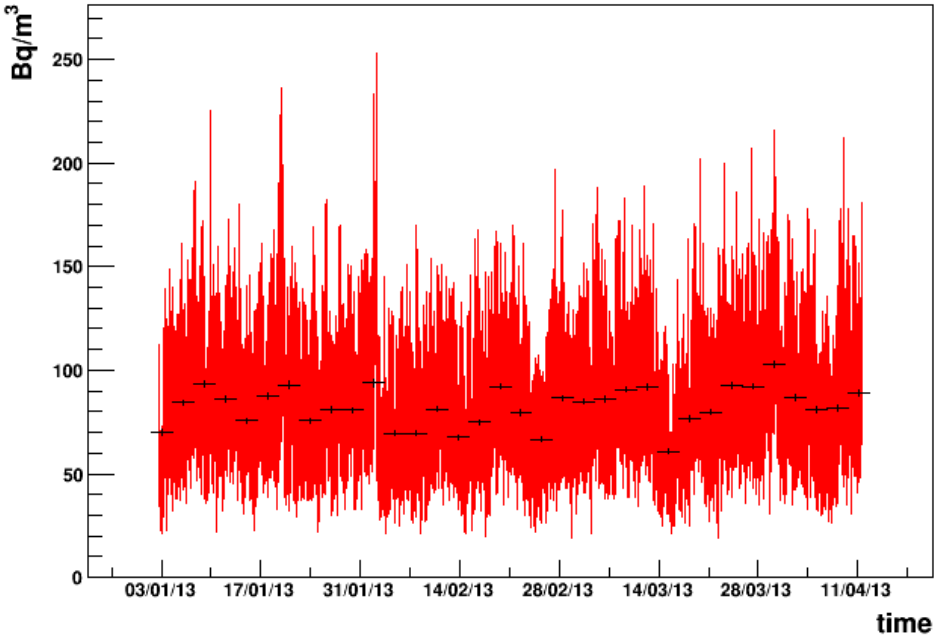
Why Rn is a problem?

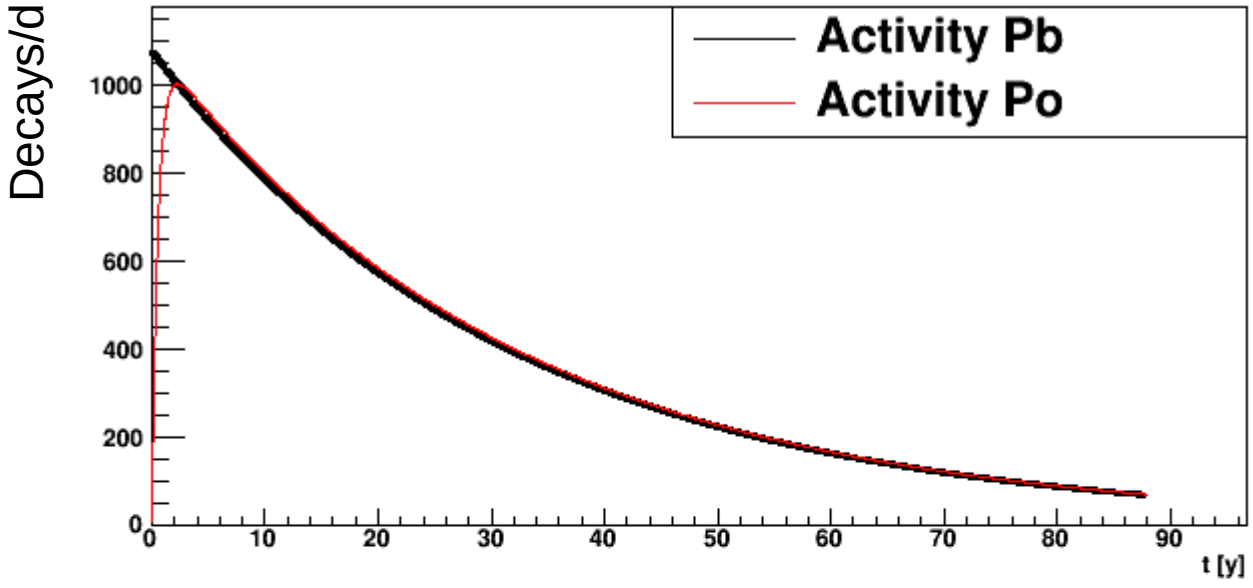
Radon is a noble gas and is able to escape both solid and liquid formations.

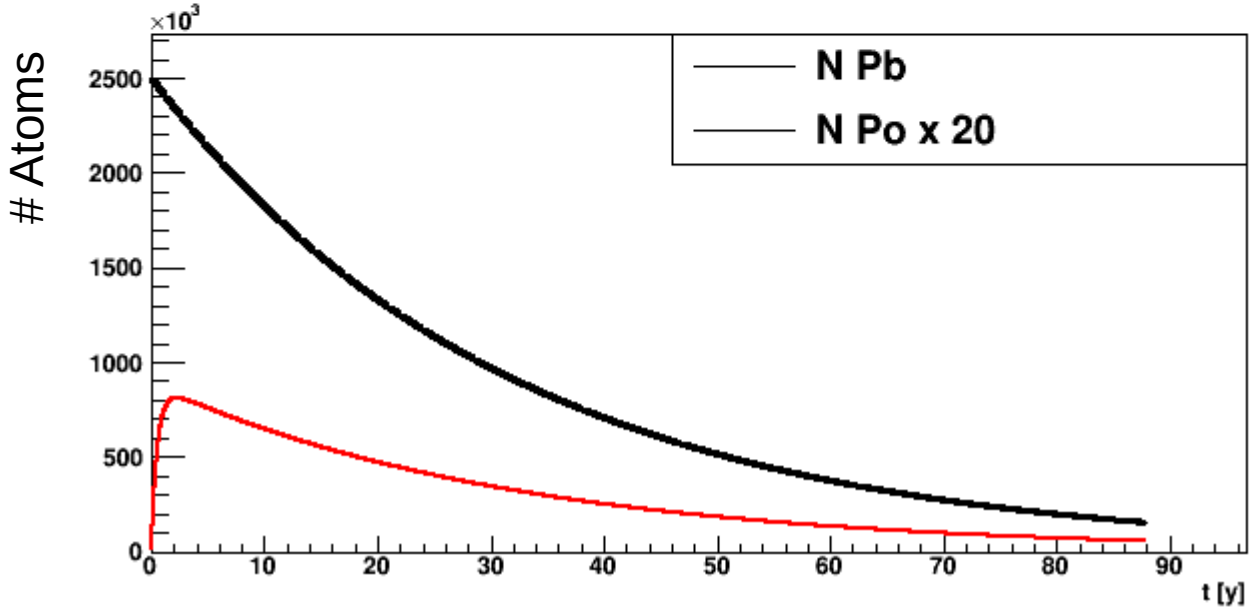
- ^{220}Rn, ^{219}Rn, and especially ^{222}Rn and their daughters release several high-energy gammas
- Rn daughters plate-out electrostatically onto surfaces. This effect is strongly enhanced on statically charged surfaces such as plastics or glass
- ^{222}Rn feeds the long-lived (22 y) isotope ^{210}Pb

Techniques based on charcoal adsorption have been used which suppress Radon (by about a factor of 10^4-10^5).

Rn is emanates constantly from the rock. ^{222}Rn concentration is significantly higher in an underground facility than on surface.

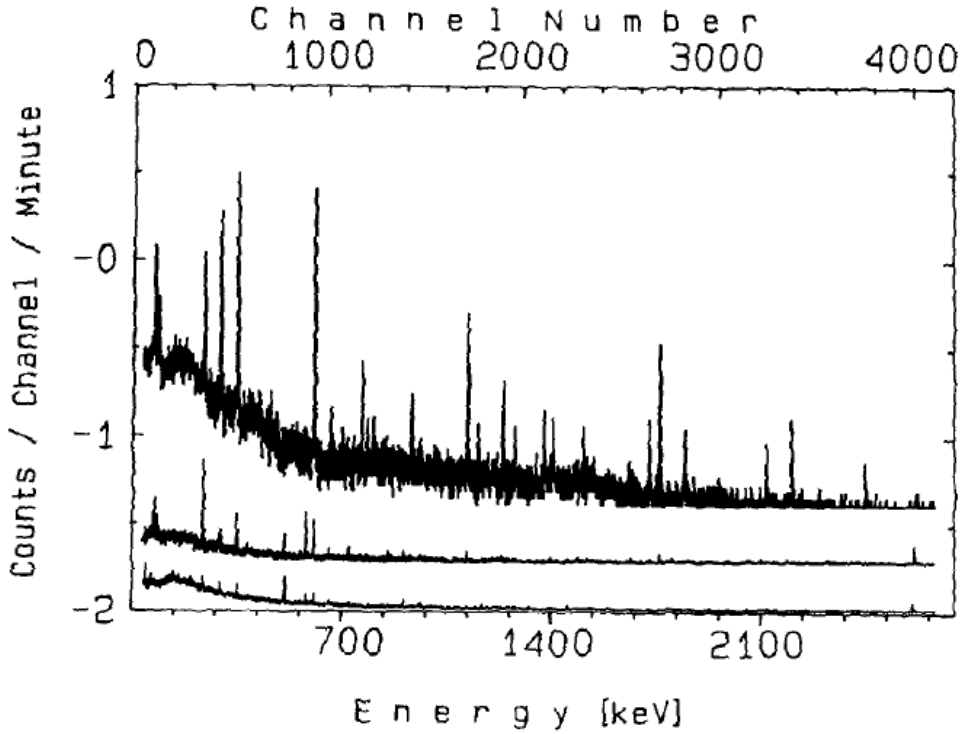






Acrylic:

1 d exposure to 135 Bq/m³



Heusser

γ Spectra of Rn progenies on plastic foils for different plate-out time
γ (~2 h, ~1 day, ~10 days)

- Surface cleaning: with acid leaching

- A small fraction of Rn can also diffuse into the plastic, which in the end also results in an hard-to-remove ^{210}Po contamination

- *It is mandatory to protect a the material surface against Rn (particularly UG)*
- *If plastic foil is used as a barrier, its permeability to Rn must be taken into account*

What we have to measure?
U, Th, K, Cs, Co…

What we have to measure?
U, Th, K, Cs, Co…

How can we measure it?

How radiopure?

1 Bq	^{238}U /kg	$\cong$ 81×10^{-9} g_U/g	(81 ppb U)
1 Bq	^{232}Th /kg	$\cong$ 246×10^{-9} g_{Th}/g	(246 ppb Th)
1 Bq	^{40}K /kg	$\cong$ 32.3×10^{-6} g_K/g	(32.3 ppm K)

- One banana is a source of about ≈ 20 Bq of ^{40}K → ≈ 100 Bq/kg
- ^{40}K in humans → primary source of radiation from our body (practically constant)

  - K content of the body is 0.2 %
  - Natural abundance of 0.0117 % of K_{Nat}
  - K-40 specific activity 2.6e5 Bq g^{-1} (using the half-life 1.28 x 10^9 y from raconv).

For a 70-kg person, the amount of ^{40}K will be about 4.26 kBq (≈ 60 Bq/kg)

- ^{14}C for a 70-kg person would be about 3.08 kBq.

radiopurity.org

Search | Submit | Edit | Settings | Login

copper

Total results: 86

Grouping	Name	Isotope	Amount	Isotope	Amount	
ILIAS UKDM	Copper	Th-232	0.004 ppb	U-238	0.005 ppb	...
ILIAS UKDM	Copper	Th-232	0.5 ppb	U-238	0.5 ppb	...
ILIAS ROSEBUD	Copper, OFHC					...
ILIAS UKDM	Copper C101	Th-232	0.5 ppb	U-238	0.5 ppb	...
ILIAS UKDM	Rexalite, copper removed	Th-232	1 ppb	U-238	3 ppb	...
EDELWEISS (2011)	Copper, Apical, cables					...
ILIAS UKDM	Copper alloy swarf	Th-232	0.5 ppb	U-238	0.5 ppb	...
ILIAS Edelweiss	Copper, Cuc2 Plate, CARLIER					...
XENON100 (2011)	Copper, Norddeutsche Affiinerie			U-238	11 mBq/kg	...
XENON100 (2011)	Copper, Norddeutsche Affinerie			U-238	70 uBq/kg	...
EDELWEISS (2011)	Copper, screens, support					...
EDELWEISS (2011)	Copper, CuC2, detector casings			U-238	1.4 mBq/kg	...
ILIAS Edelweiss	Adhesive tape, Copper					...

Common assay techniques

- Gamma counting with HPGe crystals
- Mass spectroscopy
  - ICP-MS
  - GD-MS
  - LA-ICP-MS
- Neutron Activation Analysis
- Emission spectroscopy
  - XRF
  - ICP-AES
- Radio-chemical methods (^{210}Po extraction)

Gamma spectroscopy with HPGe

- Gamma rays produced by the great majority of radionuclides
- The typical range for γ-rays emitted by nuclei is 0.1 - 10 MeV
- Gamma spectrometry is a **non-destructive technique**
- Gamma spectrometry using HPGe semi-conductor detectors is one of the best techniques to identify and quantify radionuclides: **very good energy resolution**

Material	Atomic number	Operating temperature	Band gap (eV)[a]	ε (eV)[a,b]	Density ($g\,cm^{-3}$)	Mobility($cm^2\ V^{-1}\ s^{-1}$)[a]	
						Electrons	Holes
Si	14	RT	1.106	3.62	2.33	1350	480
Ge	32	Liquid N_2 (77 K)	0.67	2.96	5.32	3.6×10^4	4.2×10^4
CdTe	48, 52	RT	1.47	4.43	6.06	1000	80
CdZnTe	48, 30, 52	RT	1.57	4.64	5.78	1000	50–80
HgI_2	80, 53	RT	2.13	4.22	6.30	100	4
GaAs	31, 33	RT	1.45	4.51	5.35	8000	400
TlBr	81, 35	−20 °C	2.68	?	7.56	—	—
PbI_2	82, 53	—	2.6	7.68	6.16	8	2
GaSe	31, 34	—	2.03	6.3	4.55	—	—
AlSb	13, 51	—	1.62	5.05	4.26	—	—
CdSe	48, 34	—	1.75	?	5.74	—	—

G.R. Gilmore

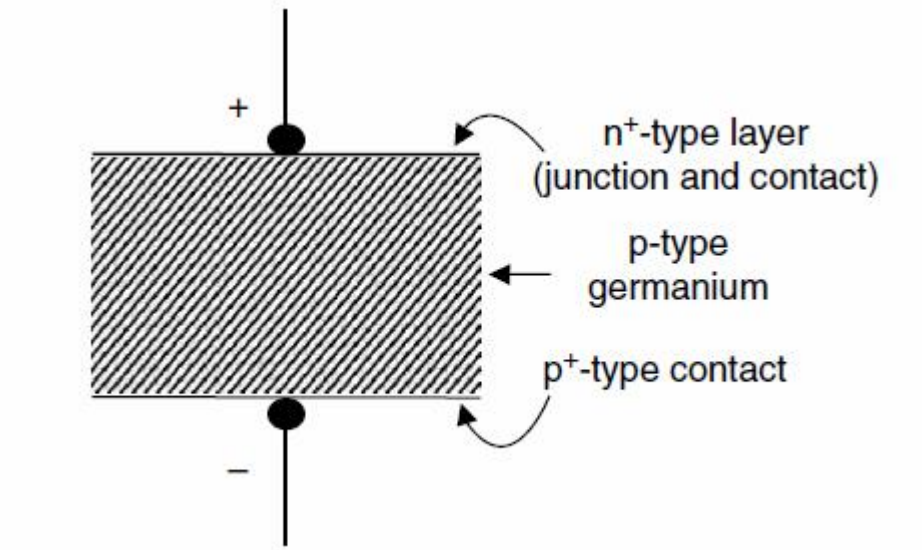
- Operating Ge detectors not possible at room temperature (leakage current due to the small bandgap energy)
- The germanium detector preamp is normally included as part of the cryostat package.
- All of the cryostat materials around the detector should be as low Z as possible to reduce photon scatter. Hence, aluminum, magnesium, beryllium, Teflon, and Mylar are used whenever possible.

Gamma spectroscopy with HPGe

Doping: Adjusting the nature of the semiconductor by adding small amounts of impurities of an appropriate type:
p-type 'p' for positive acceptor impurities; **n-type** ('n' for negative donor impurities),
→ The p-type material has an excess of holes and the n-type an excess of electrons.

The conductivity of a doped material will be higher than the intrinsic conductivity

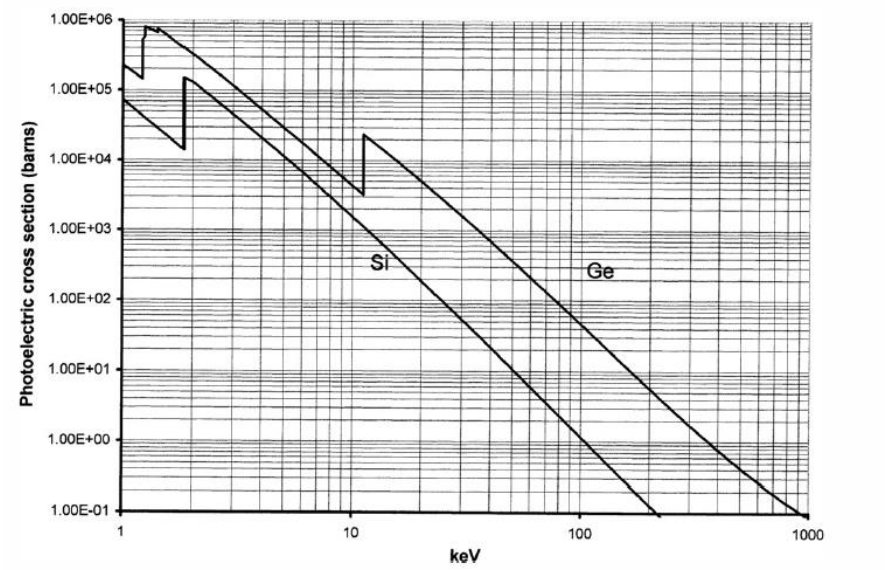
A 'standard configuration', it would be the closed-end p-type coaxial detector mounted in an aluminum outer cap



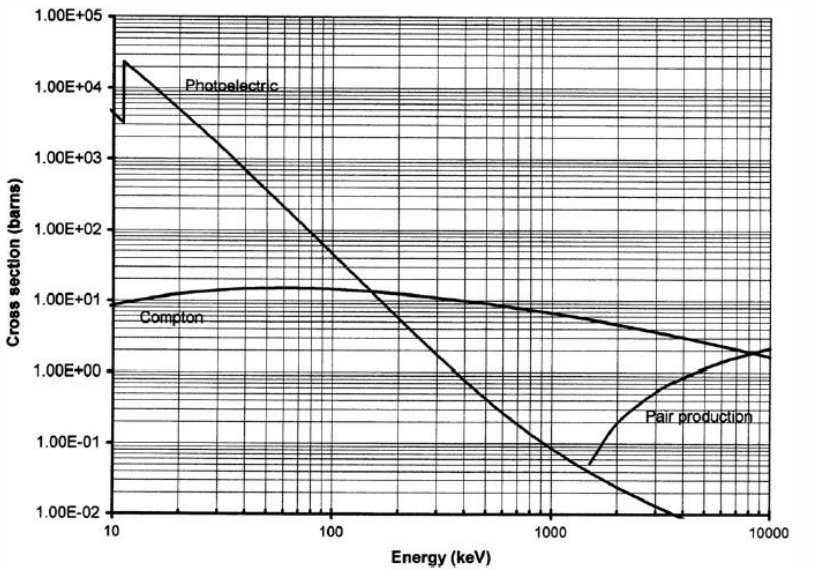
+ very high dopant concentrations produced *in situ* by evaporation or ion bombardment of appropriate impurities to produce electrical contacts for the detector

Because the positive voltage is connected to the negative type semiconductor, this is called a **reverse biased junction**

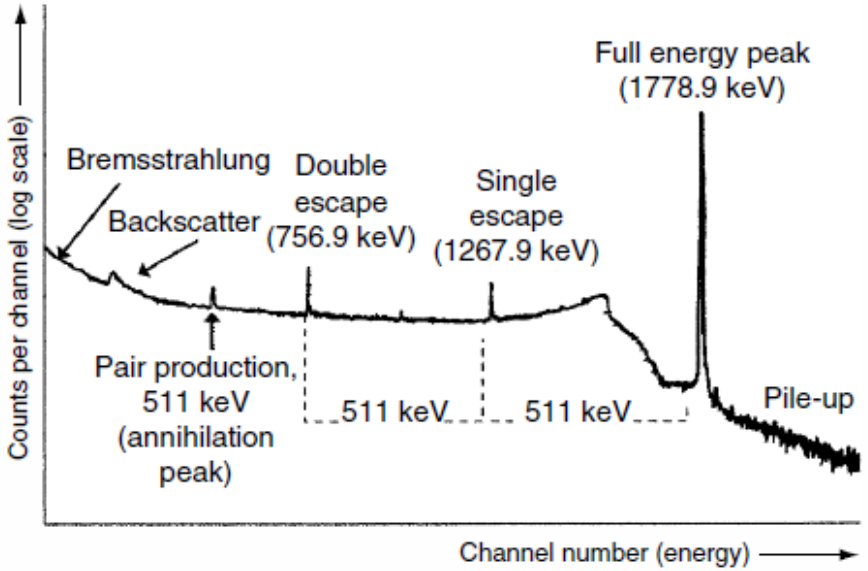
Gamma spectroscopy with HPGe



Photoelectric cross section (barns) of Si (lower curve) and Ge (higher curve) as a function of energy (keV).

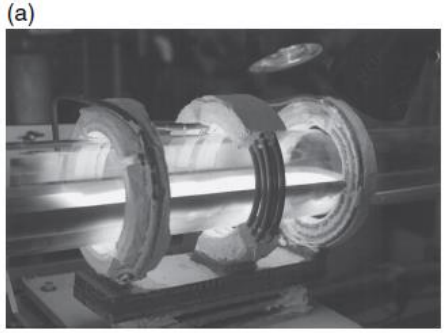


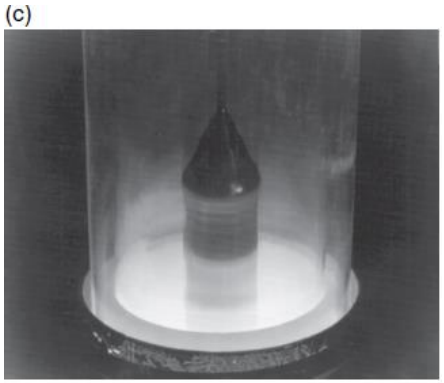
Compton, photoelectric, and pair production cross section of Ge for high-energy g-rays.



How to construct a HPGe?

Producers: ORTEC, PGT and Canberra





Stages in the manufacturing of germanium detectors:

*(a) Three RF coil zone refiner. The Ge is **melted** in a pyrolytic graphite coated quartz crucible using RF heating coils- impurities concentrate in liquid phase.*

(b) A zone refined billet of germanium.

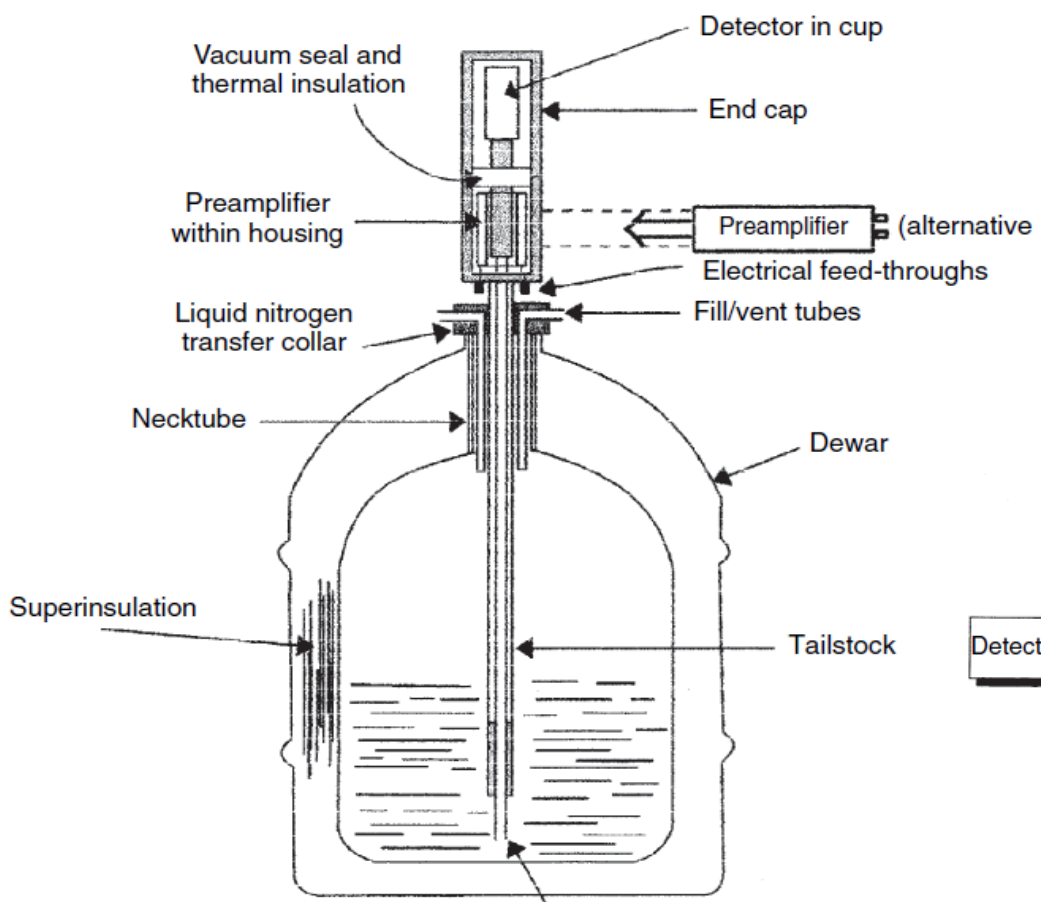
***(c) Growing** (pulling) a germanium crystal by the Czochraski technique (937 degrees Celsius and Hydrogen atmosphere).*

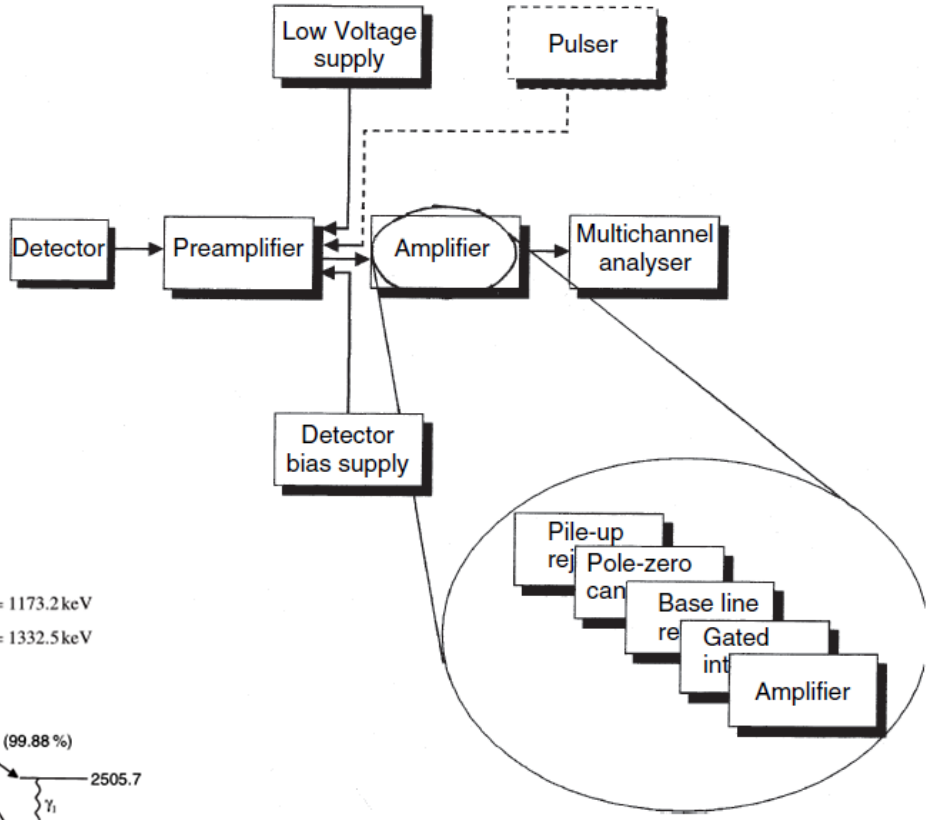
*(d) A crystal sliced by a **special string saw**.*

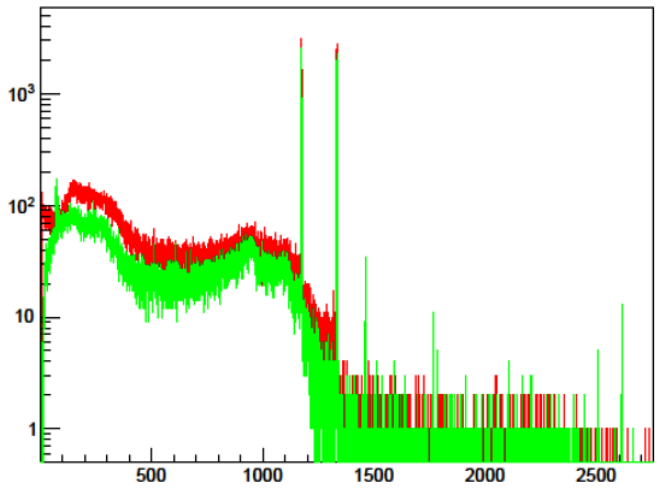
(e) Grinding the germanium crystal.

(f) Golf ball, 15% detector and 150% detector.

- The typical large detectors are cylinders approximately ~10 × 10 cm.

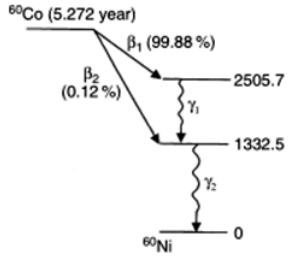


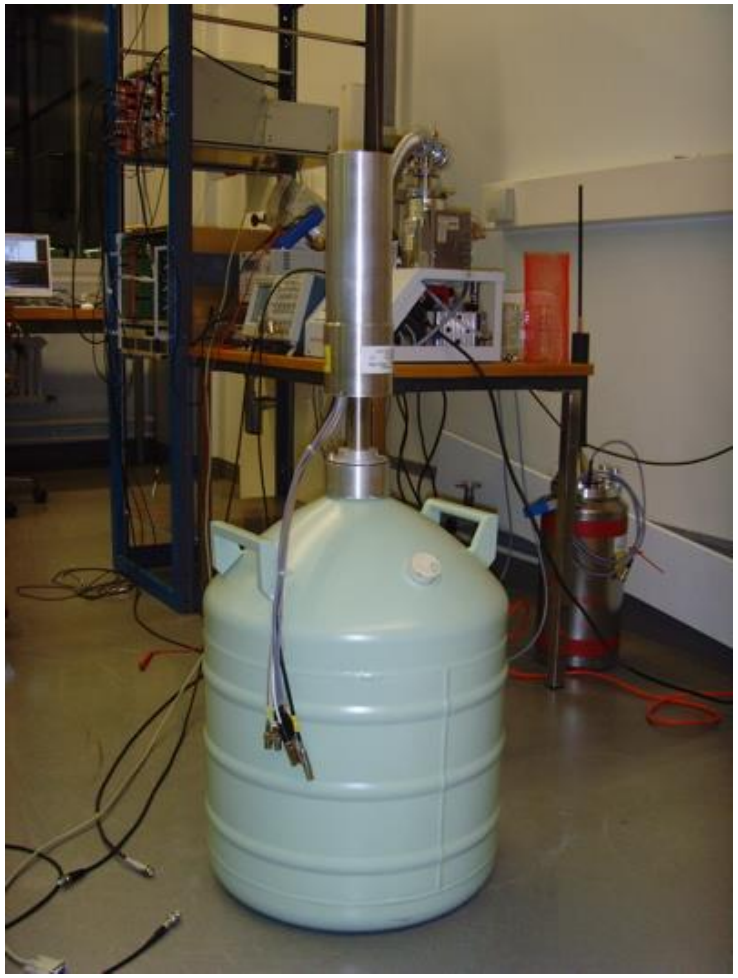


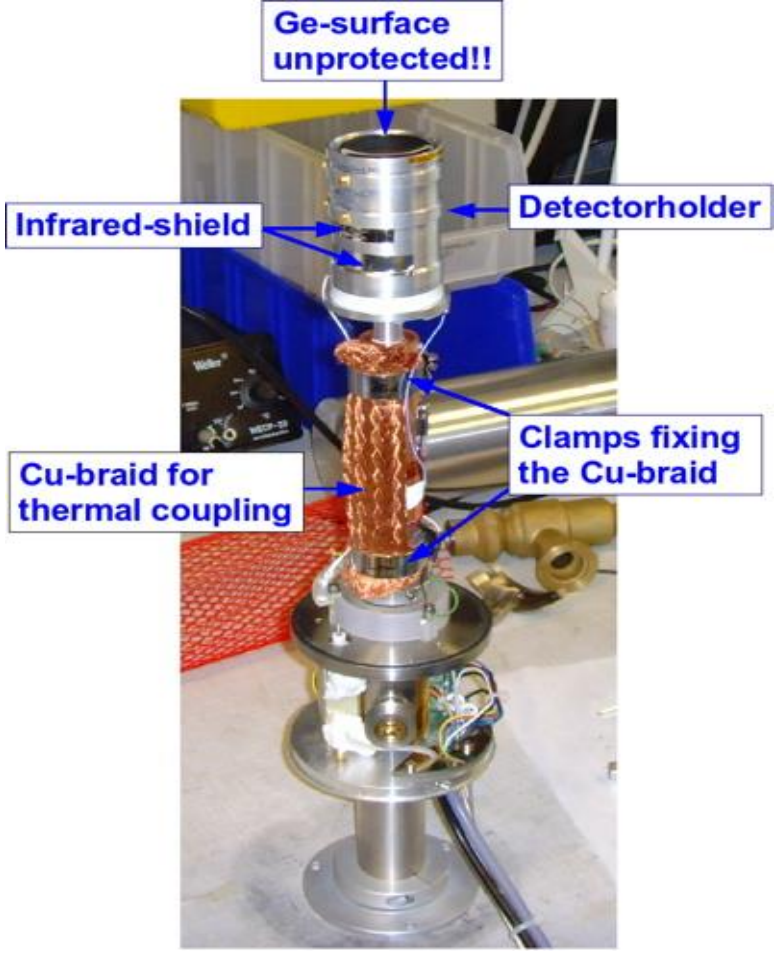


$\gamma_1 = (2505.7 - 1332.5) = 1173.2\,\text{keV}$

$\gamma_2 = (1332.5 - 0) = 1332.5\,\text{keV}$



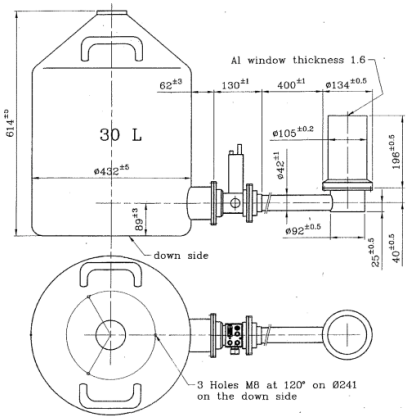




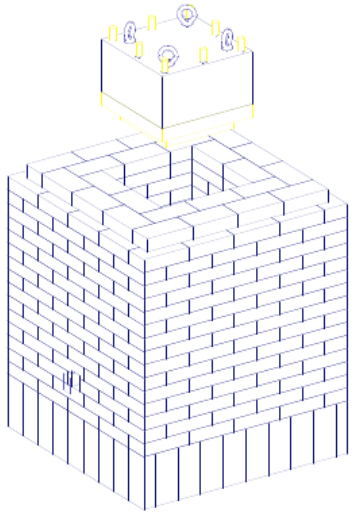
- Maximum possible efficiency of 50% due to geometry considerations.
- Well detector is basically a p-type coaxial detector with the negative contact hole drilled out large enough to fit small samples **within** the detector itself

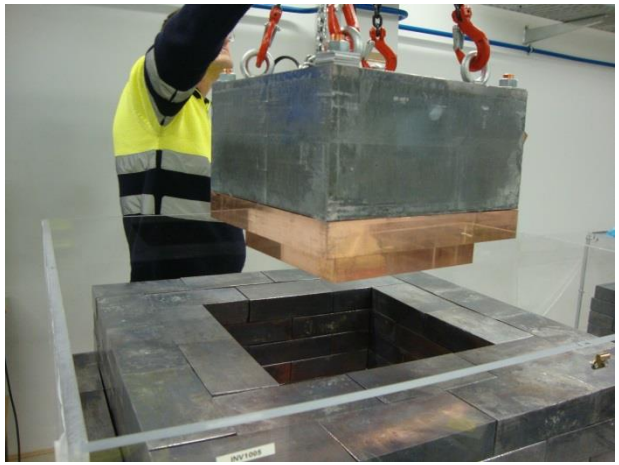
Courtesy of Iulian Bandac

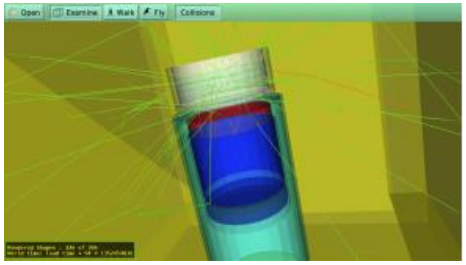
HPGe shielding

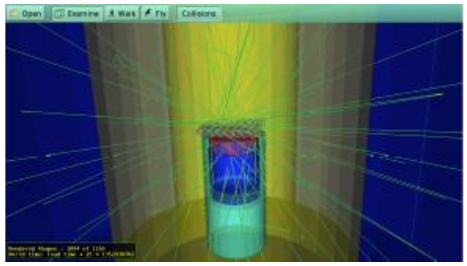


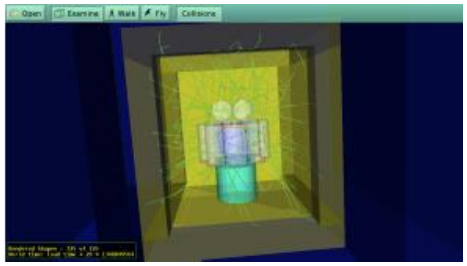
- 10 cm Cu-OF
- 20 cm Pb (~660 bricks or “donuts”)
- Methacrylate Rn box.
- Door











Courtesy of Iulian Bandac

Ultra-Low Background Measurements Service- Hall C

7 HPGe p-type coaxial (2 kg)-LSC
1 SAGe Well model GSW275L-LSC
1SAGe Well model GSW400-Poland

7 mounted and taking data: Asterix, GeLatuca, GeOroel, GeTobazo, GeAnayet, GeAspe, GeRysy.

Obelix and GeLaraca (SAGe)-no shielding

Courtesy of Iulian Bandac

LSC HPGe Characteristics

Name	V [cm3]	M [kg]	FWHM @ 1332 keV [keV]	Integral (60-2700) keV [cts/kg/day]	Tl-208 583.19 keV [cts/kg/day]	Bi-214 609.3 keV [cts/kg/day]	Co-60 1332.5 keV [cts/kg/day]	K-40 1460.8 keV [cts/kg/day]
GeOroel (p-type)	420	2.31	2.22	128.7	1.29+/-0.16	1.13+/-0.15	0.06+/-0.03	0.68+/-0.11
Asterix (p-type)	387	2.13	1.92	171.3	0.48 +/-0.08	0.73+/-0.14	0.28+/-0.06	0.62+/-0.09
GeAnayet (p-type)	410	2.26	1.99	461.2	3.68+/-0.33	0.71+/-0.14	0.16+/-0.04	0.74+/-0.08
GeLatuca (p-type)	410	2.26	1.86	305.3	3.57+/-0.33	1.16+/-0.16	0.23+/-0.05	0.56+/-0.15
GeTobazo (p-type)	410	2.26	2.02	453.8	3.35+/-0.33	0.93+/-0.16	0.23+/-0.05	0.98+/-0.15
GeAspe (p-type)	409	2.25	1.96	455.7	3.79+/-0.35	1.20+/-0.16	0.56+/-0.11	1.01+/-0.19
GeRysy* (SAGE-Well)	427	2.35	2.04	587.1	0.44+/-0.01	1.23+/-0.17	0.22+/-0.02	0.41+/-0.07

** Only 5 cm of Cu*

Gamma spectroscopy with HPGe

Acrylic sample

	A (mBq/kg)
U-235/U-235	< 0.44
U-238/Pa-234m	< 34
U-238/Ra-226	< 1.2
Th-232/Ac-228	< 1.4
Th-232/Th-228	< 0.71
K-40	< 4.6
Cs-137	< 0.22
Co-60	< 0.22

Acrylic sample

Parent	Daughter	Energy/keV	Efficiency/%	Specific Activity mBq/kg
U238_E	Th234	63.3	19.4	< 6.3
U238_E	Th234	92.5	18	< 9.2
U238_L	Pb214	295.2	8.8	< 11.1
U238_L	Pb214	351.9	7.4	< 5.9
U238_L	Bi214	609.3	4.4	< 9.7
Pb210	Pb210	46.5	19.4	< 9.2
Th232_E	Ac228	338.3	7.7	< 5.7
Th232_E	Ac228	911.2	3	< 10.8
Th232_E	Ac228	969	2.9	< 12.1
Th232_L	Pb212	238.6	10.9	< 1.8
Th232_L	Tl208	583.2	4.6	< 4.2
Th232_L	Tl208	2614.5	1.3	< 7.9
K40	K40	1460.8	2.1	65 ± 32
U235	U235	143.8	15.9	< 0.3
Co60	Co60	1173.2	2.5	< 1.9
Co60	Co60	1332.5	2.3	< 2.1
Cs137	Cs137	661.7	5.6	< 0.3

HPGe: Figure of Merit

Advantages:

- The best technique to assay the γ-emitters: K-40, middle primordial decay chains, cosmogenics … etc
- Relatively simple sample preparation
- Non-destructive technique
- Very good sensitivity achievable (≈ mBq/kg)

Limitations:

- Actual sensitivity strongly depends on the mass of the sample (not suitable for small samples)
- Stringent detector stability and background requirements
- Only sensitive to γ-emitters
- Several weeks are typically necessary to assay clean samples
- Detailed Monte Carlo simulation to calculate the efficiency as a function of E

MASS SPECTROMETRY (MS)

- Inductively coupled plasma mass spectrometry (ICP-MS)
- Glow discharge mass spectrometry (GD-MS)
- Laser ablation mass spectrometry (LA-ICP-MS)
- Secondary ion mass spectrometry (SIMS),
- Resonance ionization mass spectrometry (RIMS),
- Accelerator mass spectrometry (AMS)

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- *Accelerator mass spectrometry (AMS)*

INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY (ICP-MS)

It allows the determination of **elements** with atomic mass ranges 7 to 250 (Li to U).

Concentration at very low level in a wide variety of samples.

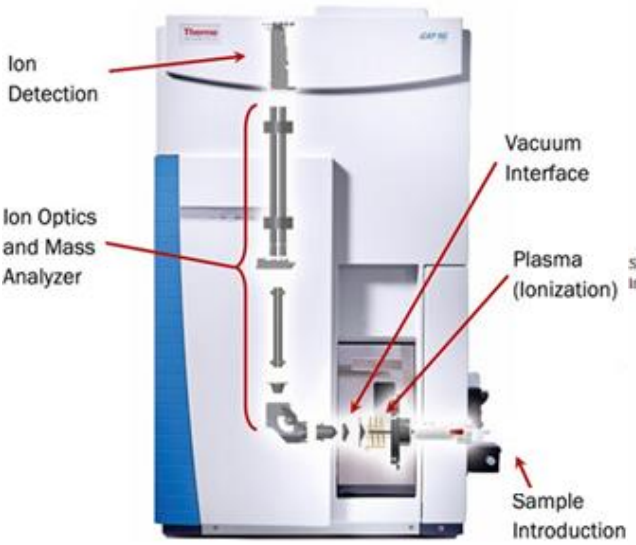
High resolution ICP-MS (HR-ICP-MS), uses a magnetic field and electrostatic analyzer, providing very low detection limits, in the $pg \cdot L^{-1}$ range.

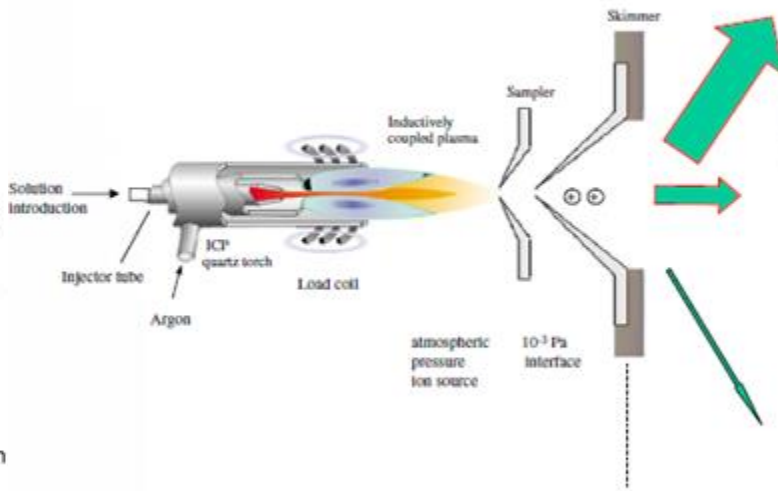
Destructive analysis: a **previous sample digestion** is required in order to obtain a liquid solution.

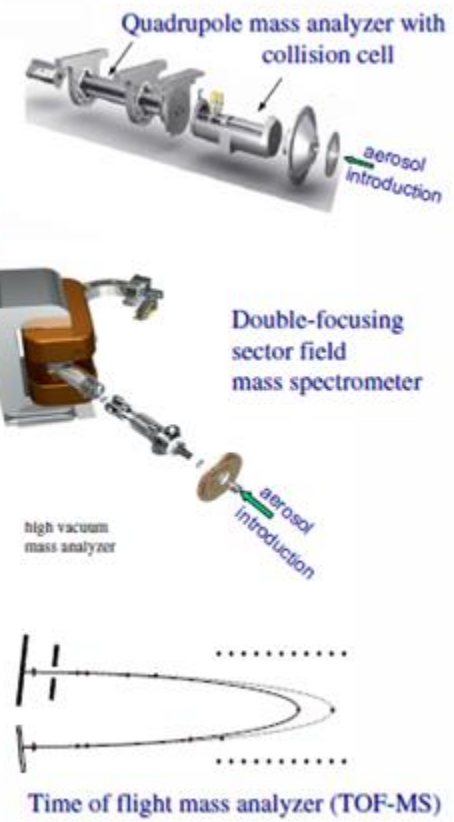
In comparison to other alternative solid-state mass spectrometric techniques is advantageous because liquid solutions can be easily and rapidly analyzed.

Major advantage of ICP-MS: the ICP source is an atmospheric ionization source in which the nebulized solution or the ablated material is introduced directly under atmospheric pressure.

INDUCTIVELY COUPLED PLASMA MASS SPECTROMETRY (ICP-MS)





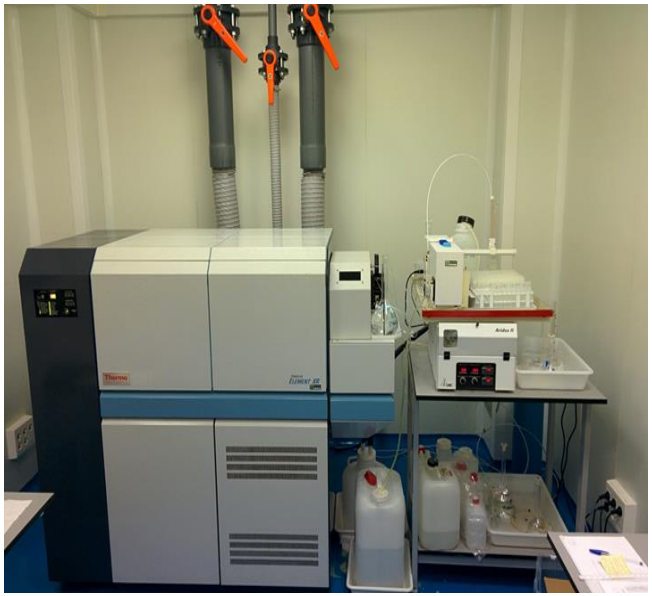


Perkin, Agilent, Bruker….

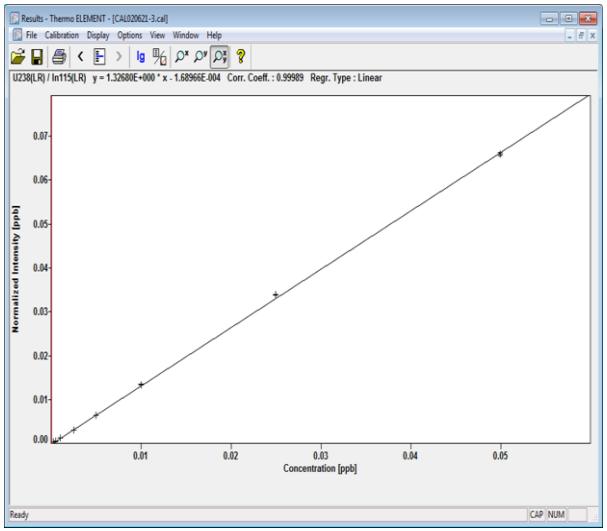
- **ICP source**
- Extraction from the atmospheric pressure using an **ion lens** system
- ICP **low-pressure interface region to the high vacuum** of the mass spectrometer
- Three types of **analyzers**

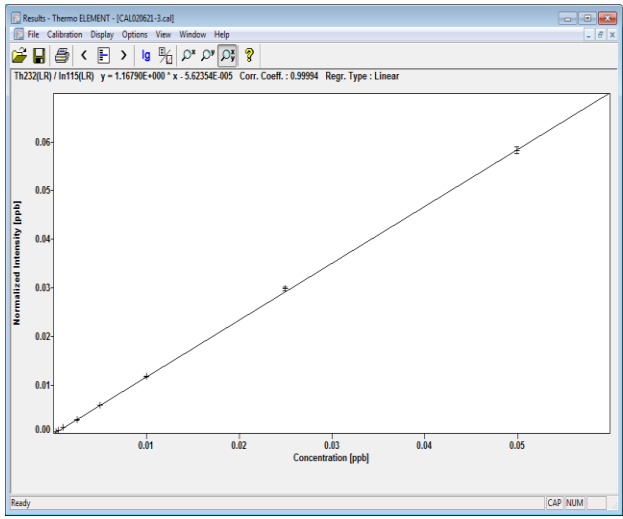
ICP-MS

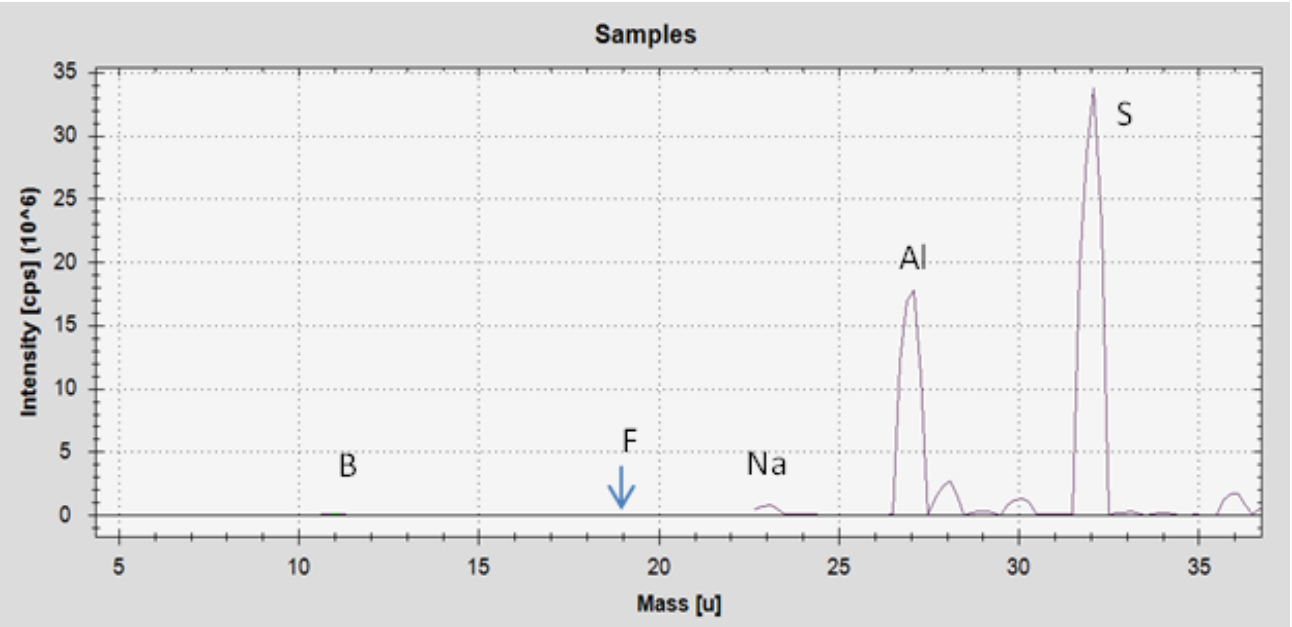
HR-ICP-MS Thermo Element



^{238}U AND ^{232}Th CALIBRATION

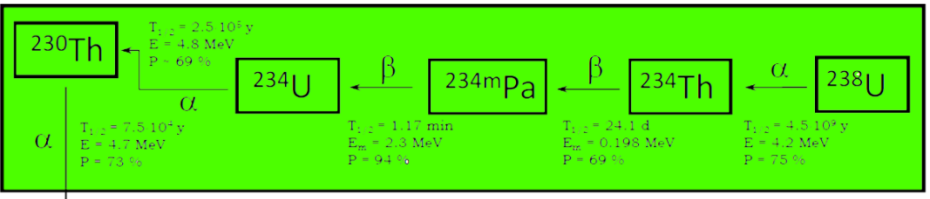


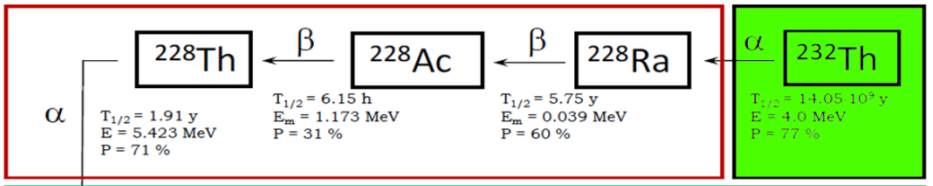


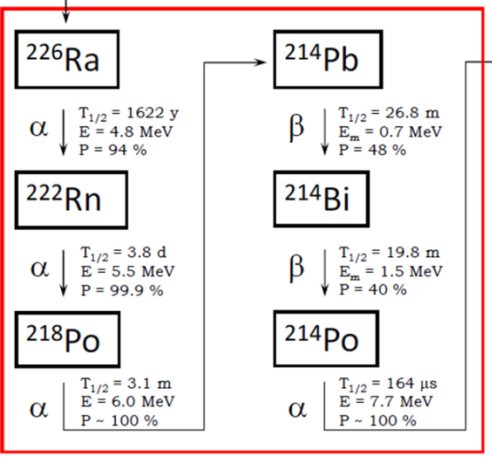


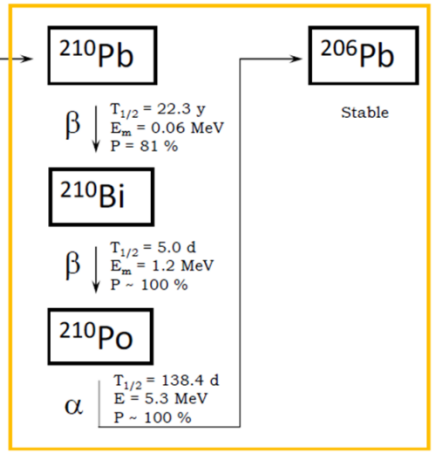
	Th (ng/g)	U (ng/g)
LOQ	0.052	0.580

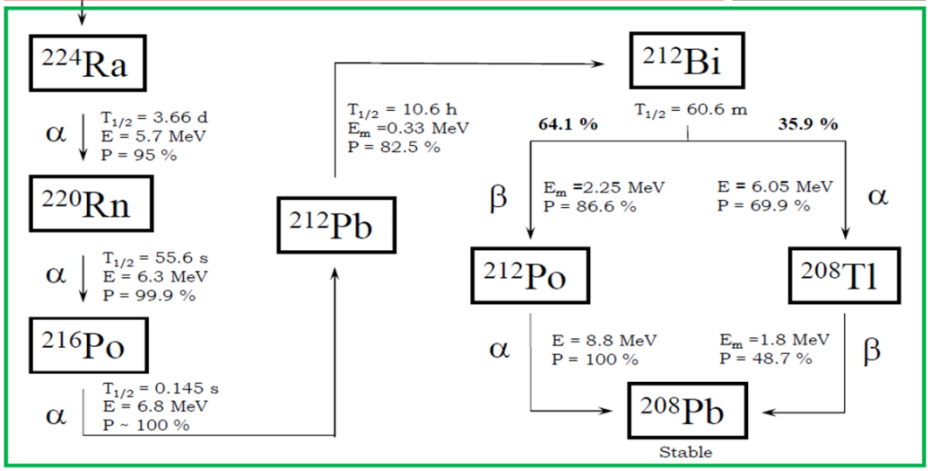
Spectroscopic interferences (molecular ion formation) can be decreased or overcome by the use of a collision cell.











---------- Summary ----------

Your input : 0,000001 mg 238Uc

ID	NUCL	Nb. Atoms	Ac[Bq]	Mass[g]
16	**238U**	**2,529772e+12**	**1,243652e-05**	**1,000000e-09**
17	234Th	3,735978e+01	1,243652e-05	1,451975e-20
18	234Pa	1,264919e-03	1,243652e-05	4,915961e-25
19	**234U**	**1,387185e+08**	**1,243652e-05**	**5,391072e-14**
20	**230Th**	**4,267999e+07**	**1,243652e-05**	**1,630286e-14**
21	**226Ra**	**9,059165e+05**	**1,243652e-05**	**3,400122e-16**
22	**222Rn**	**5,927183e+00**	**1,243652e-05**	**2,185167e-21**
23	**218Po**	**3,283404e-03**	**1,243652e-05**	**1,188633e-24**
24	**218At**	**6,459207e-09**	**2,238574e-09**	**2,338310e-30**
25	214Pb	2,884570e-02	1,243428e-05	1,025046e-23
26	**214Bi**	**2,142289e-02**	**1,243652e-05**	**7,612691e-24**
27	210Tl	2,938889e-07	2,611670e-09	1,024781e-28
28	**214Po**	**2,947269e-09**	**1,243391e-05**	**1,047304e-30**
29	210Pb	1,262621e+04	1,243652e-05	4,402594e-18
30	210Bi	7,771144e+00	1,243652e-05	2,709695e-21
31	**210Po**	**2,145474e+02**	**1,243652e-05**	**7,480941e-20**

Total

1,741113e-04	1,000071e-09
1,119283e-04 (a)	
6,218299e-05 (b)	

---------- Summary ----------

Your input : 0,000001 mg 232Thc

ID	NUCL	Nb. Atoms	Ac[Bq]	Mass[g]
5	**232Th**	**2,595325e+12**	**4,057385e-06**	**1,000000e-09**
6	228Ra	1,062144e+03	4,057385e-06	4,021854e-19
7	228Ac	1,296841e-01	4,057385e-06	4,910543e-23
8	**228Th**	**3,531120e+02**	**4,057385e-06**	**1,337061e-19**
9	**224Ra**	**1,852267e+00**	**4,057385e-06**	**6,890321e-22**
10	**220Rn**	**3,256625e-04**	**4,057385e-06**	**1,189765e-25**
11	**216Po**	**8,493271e-07**	**4,057385e-06**	**3,046363e-28**
12	**212Pb**	**2,243639e-01**	**4,057385e-06**	**7,898076e-23**
13	**212Bi**	**2,128074e-02**	**4,057385e-06**	**7,491241e-24**
14	208Tl	3,856248e-04	1,458224e-06	1,331802e-25
15	**212Po**	**1,121930e-12**	**2,599161e-06**	**3,949369e-34**

Total

4,057385e-05	1,000000e-09
3,100085e-05 (a)	
9,572994e-06 (b)	

Concentration of the daughters too low for MS

SAMPLE DIGESTION FOR ICP-MS

- ACID DIGESTION METHODS

Acid digestion uses a combination of different acids in order to obtain a total simple digestion (strong mineral acids: Nitric Acid, Hydrochloric Acid, Hydrofluoric Acid, Perchloryc Acid…)

Hydrofluoric acid (HF) is the most effective mineral acid for breaking up strong Si–O bonds to form silicon hexafluoride ions (F_6^{2-})in acidic solution. Disadvantages:

Silicates are converted to volatile Silicon Tetrafluoride (SiF_4), which will be lost in open vessel digestion procedures.

To analyze boron in samples that require HF to be digested, glassware should be avoided, because with HF borosilicates are released, producing contamination.

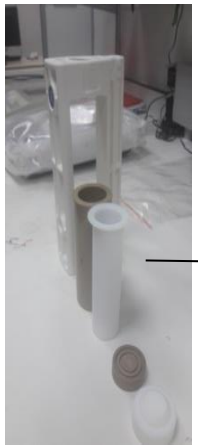
OPEN VESSEL ACID DIGESTION

Acid attack in open containers placed on a hot plate.

MICROWAVE ACID DIGESTION

Performed in closed vessel bombs with high temperature and pressure conditions.

sample + Acids mixture



Microwave oven

DISOLUTION IN HOT PLATE

HNO_3 + HCl mixture

+ *Solution for ICP-MS analysis - 1*

+ HF

Solution for ICP-MS analysis - 2

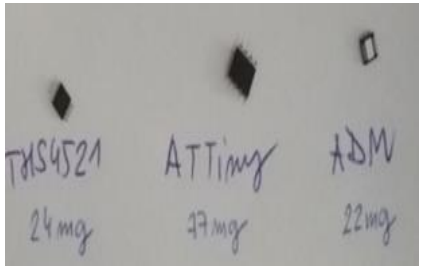
DISOLUTION IN MICROWAVE

Polymeric sheets

Solution for ICP-MS analysis - 1

+ HF

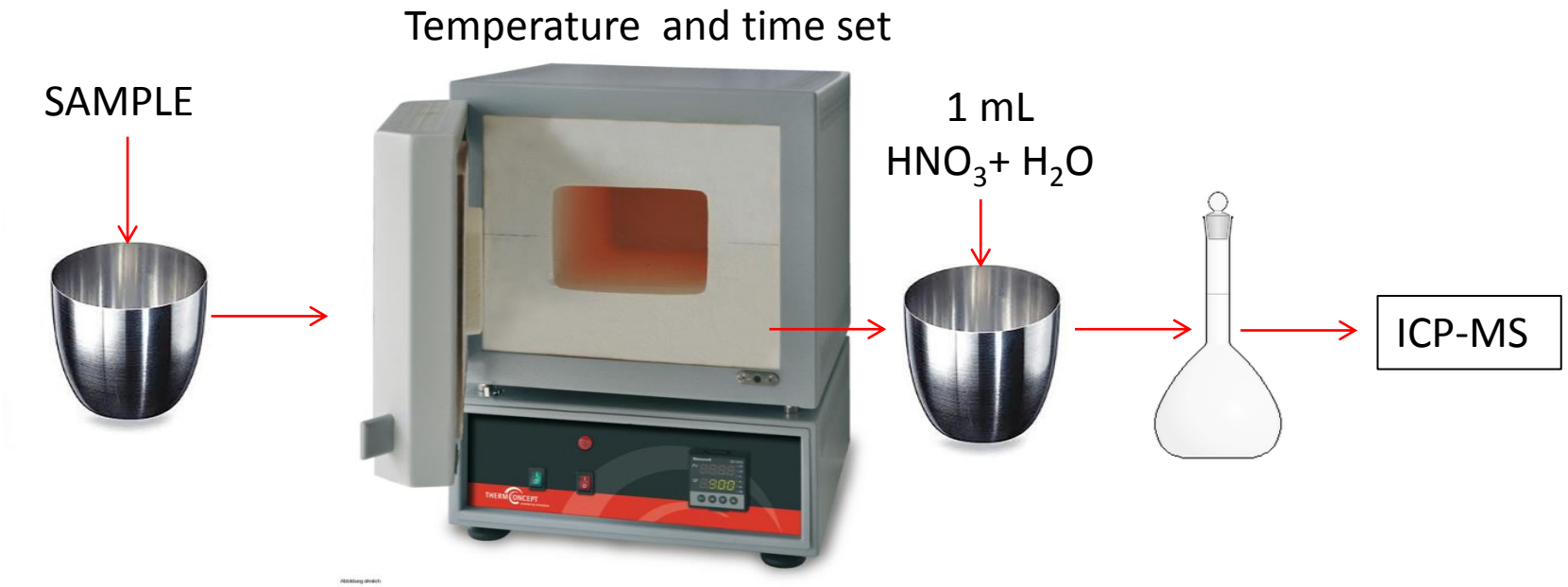
Solution for ICP-MS analysis - 2



Electronic components

Solution for ICP-MS analysis

SAMPLE DIGESTION: Dry ashing



- The organic matter can be completely eliminated.
- The best vessel is platinum crucible (otherwise quartz) which is unaffected by any of the usual acids, and it does not induce contamination.
- Especially suitable for samples with a high organic matter content.

SS sample

Sample preparation

The samples were etched in three phases with 6 ml of ultra pure Hydrochloric acid and 2 ml ultra pure nitric acid. Then the samples were diluted 20 times before ICP-MS measurements

The first etching has been considered as cleaning but however has been measured to look for superficial contamination informations. For samples N 4 and 5 was difficult to control the reaction that was quite violent.

Sample.Ecth	Initial weight [g]	Final weight [g]	Difference [g]	Volume [ml]	Dilution
1.1	13.81	12.28	1.53	15	9.8
2.1	13.84	12.28	1.56	10.5	6.7
3.1	4	2.5	1.5	12.5	8.3
4.1	10.6	9.28	1.32	12.5	9.5
5.1	9.95	8.36	1.59	17	10.7
1.2	12.28	10.81	1.47	17.5	11.9
2.2	12.28	10.78	1.5	15	10.0
3.2	2.5	1.53	0.97	11	11.3
4.2	9.28	8.73	0.55	14	25.5
5.2	8.36	7.61	0.75	15	20.0
1.3	10.81	9	1.81	18	9.9
2.3	10.78	9.11	1.67	19	11.4
3.3	1.53	0	1.53	16	10.5
4.3	8.73	5.93	2.8	12.5	4.5
5.3	7.61	5	2.61	20	7.7

		Reagents
Sample 1	SS-8-VCR-4	2.0 ml HNO3+6.0 ml HCl
Sample 2	SS-8-VCR-3	2.0 ml HNO3+6.0 ml HCl
Sample 3	Flex pipe small	2.0 ml HNO3+6.0 ml HCl
Sample 4	Flex pipe medium	2.0 ml HNO3+6.0 ml HCl
Sample 5	Flex pipe big	2.0 ml HNO3+6.0 ml HCl

Analysis results

The concentrations were evaluated in semiquantitative analysis, based on the response of a single spike solution containing 100 ppt of Th and U added to aliquot of Step 3 . The uncertainty is about 30% of the given values. The contribute of procedure and reagents has been subtracted to the samples.

	Pb	Th	U
	[ng/g]	[ng/g]	[ng/g]
1.1	280	1.4	2.8
2.1	480	1.9	7.0
3.1	455	1.5	6.1
4.1	620	2.5	9.1
5.1	510	2.8	9.6

Table: 1st etching cleaning step measurements

ICP-MS: Figure of Merit

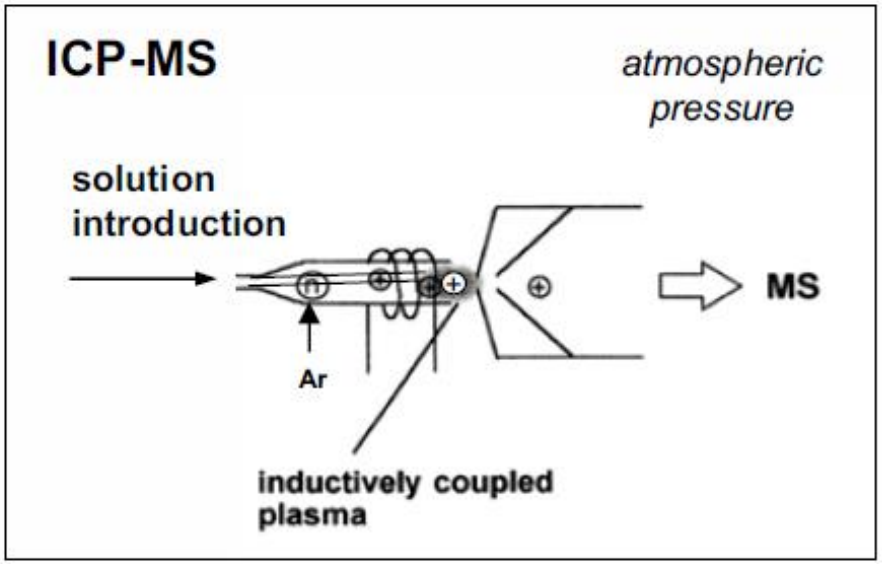
Advantages:

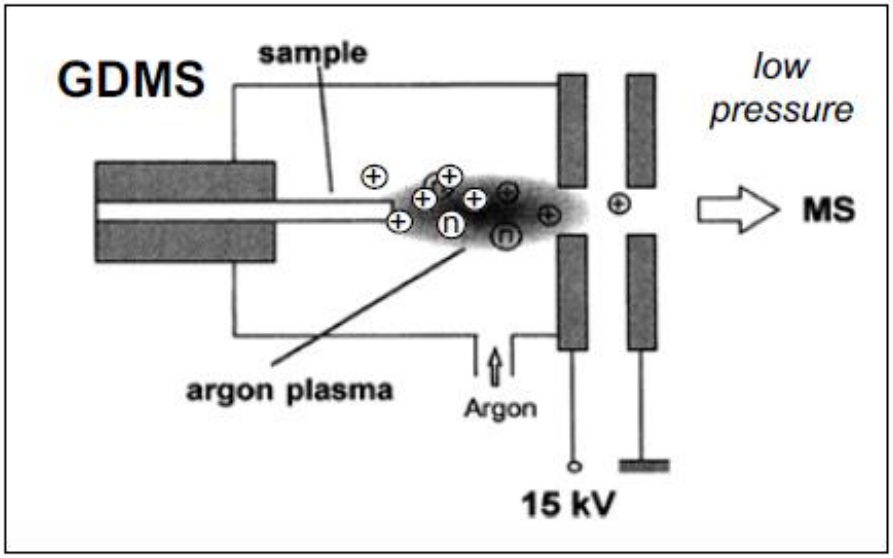
- **Fast analytical technique for simultaneous determination of trace and ultra-trace elements in liquid solution (≈ 1 d)**
- **Excellent sensitivity, accuracy and very low det. limits: ~ 0.1 pg/mL**
- **Little amount of materials needed (small sample)**
- **Isotope ratio measurements**

Limitations:

- Destructive analysis
- Sample preparation for solid samples (full digestion, recuperation, contamination..)
- It only measures the upper chain (It cannot measure K-40)
- Molecular ions interferences with atomic ions (e.g. $^{238}UH^{+}$ and $^{239}Pu^{+}$)

Ion sources in mass spectrometry for long lived radionuclide analysis





It allows a fast analysis of (mostly conducting) solids

An argon gas glow discharge (GD) at a pressure of 0.1 - 10 torr is used as an ion source

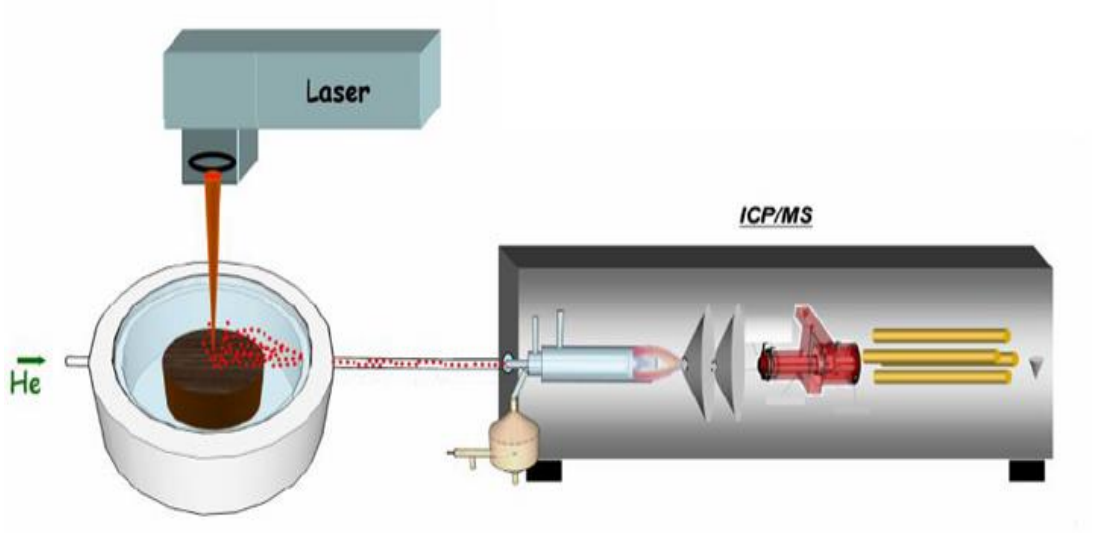
The sample material (cathode surface) is sputtered by Ar^+ ions, which are formed in low-pressure argon plasma and accelerated toward the cathode

Sputtered neutral particles of sample are ionized in the glow discharge plasma

LA-ICP-MS

Concept: to replace the chemical preparation of a sample by using a laser in ablation mode

- ***Avoid time consuming sample preparation***
- ***Small risk of sample contamination***



- Focused laser beam (e.g. **neodymium-doped yttrium aluminum garnet** laser) in an argon atmosphere at normal pressure.
- Mostly quadrupole mass spectrometers, are applied (interference problem similar to ICP-MS)
- The limits of detection (LODs) for the determination of trace metals (bulk analysis) depend on the laser parameters, laser power density, wavelength of laser beam, the matrix composition.

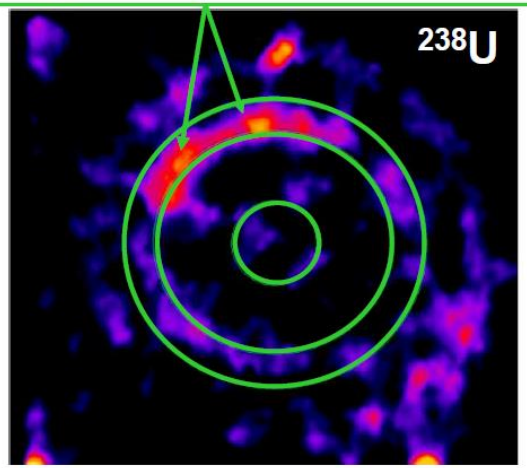
Short laser pulse duration $\approx 10^{-13}$ s

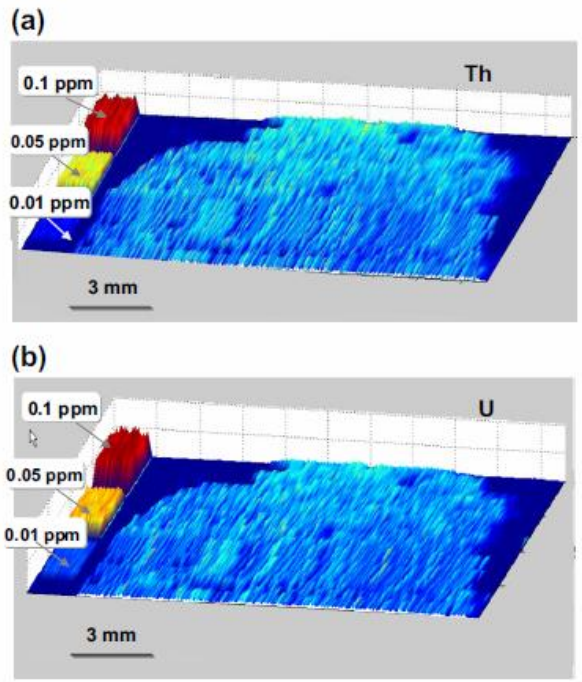
- Little thermal effects
- Less systematics effects (small size of particles, negligible rediposition)
- High depth resolution
- Uncertainties on depth/volume ablated
- Size of the beam ~10 µm
- High repetition rate ~100 kHz
- Translation speed < 2m/s

Imaging with LA-ICP-MS

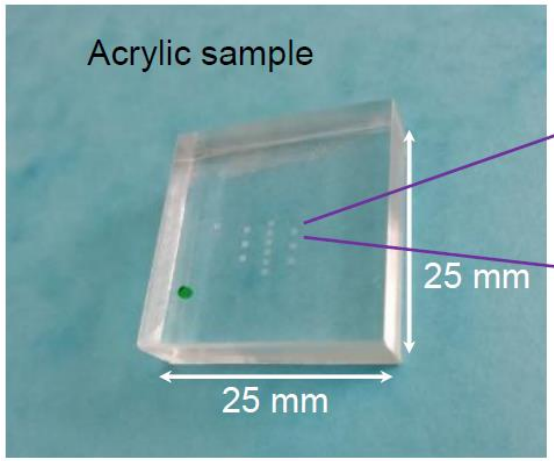
F. Perrot talk LRT-2019, Jaca

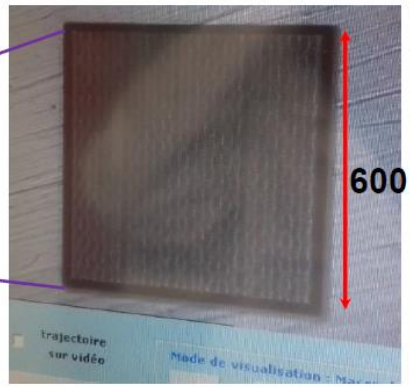
U contamination observed in some of the copper wires !





Quantitative images of (a) Th and (b) U (10 ng·g^{-1} concentration) in biological tissue





2D scan of a square of 600x600 µm^2 with a laser spot of 8 µm

LA-ICP-MS: drawback for trace analysis

The mass of ablated sample depends on:

- the laser: energy, wavelength, duration of the pulse, rate of laser shots, beam diameter
- **the sample**: optical, mechanical and thermal properties
  $\rightarrow$ *this is difficult to parametrize*

Main drawback for trace analysis: need for a suitable **reference material** in order to quantify analytical data

"Homemade" calibration with reference materials similar to the sample of interest

Complementarity of the techniques:

- LA-ICP-MS able to identify where the contamination comes from
- Classical ICP-MS able to measure it quantitatively

LA-ICP-MS: Figure of Merit

Advantages:

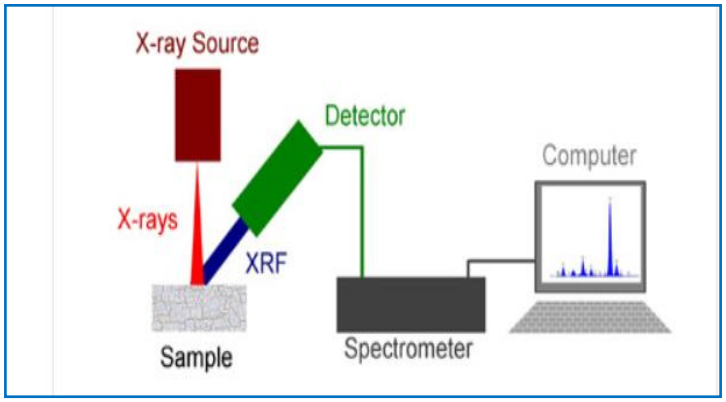
- **No chemical preparation needed**
- **Nearly non-destructive technique (negligible amount of material needed)**
- **It can be very fast (< 1d)**
- **Spatial information for micro-analysis**
- **Low LoD (similar to ICP-MS)**

Limitations:

- It only measures the upper chain (It cannot measure K-40)
- Molecular ion formation/Interferences with atomic ions (e.g. $^{238}UH^{+}$ and $^{239}Pu^{+}$)
- Need standard reference materials with similar matrix composition

Emission spectroscopy

- X-Ray fluorescence (XRF)



- N hnique
- Sa -ray sc
- M ondary) X-ra each sp

This a wide rang (F) to urani

- Inductively coupled plasma - atomic emission spectroscopy ICP-AES

- ICP and the optical spectrometer
- Ar gas is typically used to create the plasma
- Molecules break up into their respective atoms which then lose electrons and recombine repeatedly in the plasma,
- Radiation at the characteristic wavelengths of the elements is produced

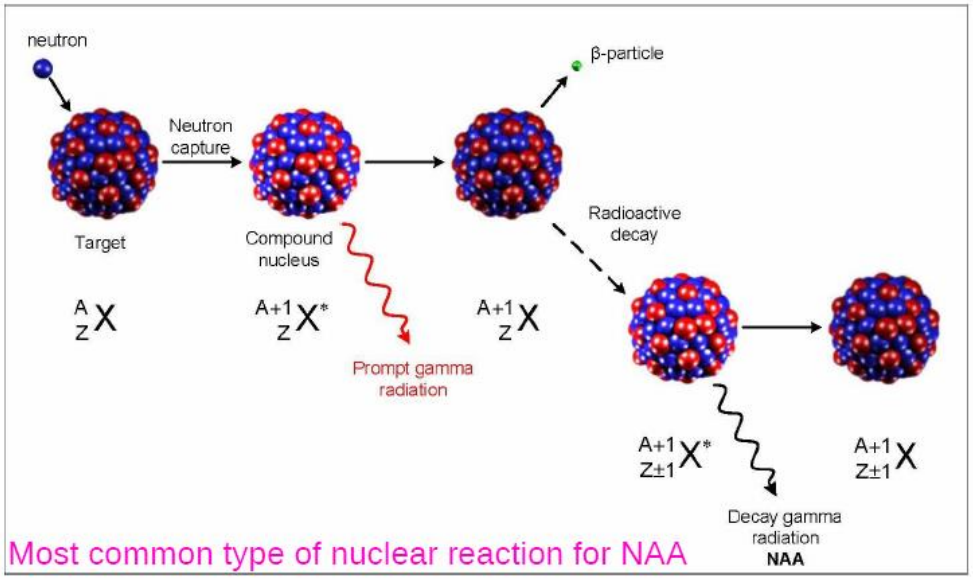
Resultados	Referencia	Al (%)	Si (%)	Ni (%)	P (%)	S (%)	Ca (%)	Fe (%)	Cu (%)	Ag (%)	Sn (%)	Au (%)	Bi (%)	As (%)	Ru (%)	Pd (%)
Muestra 7	ATTINY102		91,00		0,34	1,70			0,78	5,80						
Muestra 8	ADM7150ACPZ		81,00		0,09	0,92		0,66	1,50	15,00		0,48	0,86			
Muestra 9	THS4521		57,00	6,00					35,00	1,30						
Muestra 10	OPA838		46,00	6,30				1,00	47,00							
Muestra 12	Diode BAP65		3,50	5,30					51,00	17,00	23,00					
Muestra 13	tantalum 33uF F381A33		0,85	40,00	0,95		1,90		48,00	5,20		2,30				
Muestra 14	5 Ohm resistors 716-853	5,90	8,00	24,00	0,08				0,44	11,00	45,00		6,00	0,22	0,38	0,42

CONCENTRATION RANGE % BY WEIGHT

Neutron Activation Analysis (NAA)

It allows the precise identification and quantification of the elements. For many elements and applications, NAA offers sensitivities that are superior to those attainable with other methods.

Basic concept: This method is based on conversion of stable atomic nuclei into radioactive nuclei by irradiation with neutrons and subsequent detection and identification of the radiation emitted by the radioactive nuclei



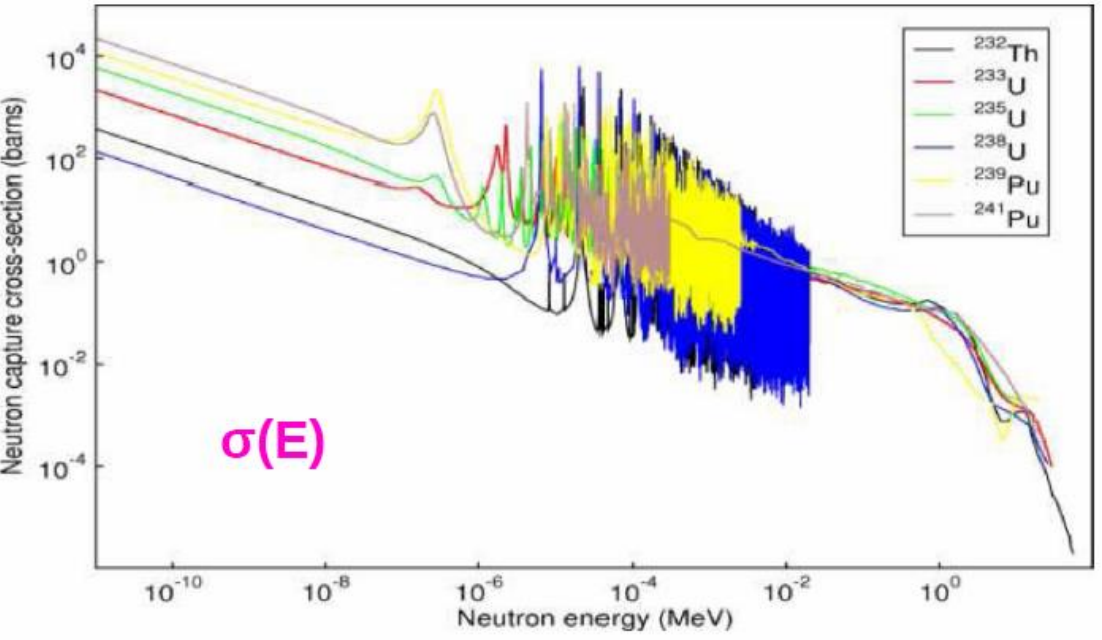
- Non-elastic collision.
- The compound nucleus de-excite almost instantaneously ($\sim 10^{-14}$ s)
  $\rightarrow$ emission of one or more characteristic prompt gamma rays: PGA
- In many cases the nucleus decays (with its specific $t_{1/2}$)
  $\rightarrow$ characteristic delayed gamma rays: DGA

- Source of neutrons
- γ-ray detectors
- Detailed knowledge of the neutron capture reactions

Neutron Activation Analysis (NAA)

- DGNAA is the most common NAA method
- The PGNAA technique is mostly applicable to elements with extremely high neutron capture cross-sections (B, Cd, Sm, and Gd)
- Facilities with water moderated research nuclear reactor typically used (10^{11} - 10^{14} n·cm^{-2}·s^{-1})

Neutron capture cross section vs Energy for major actinides



Activation rate

$R = N \times \sigma_{eff} \times \Theta_{Tot}$

$N \rightarrow$ amount of the original, stable isotope in the sample

$\sigma_{eff} \rightarrow$ *effective cross section,* (mean value of x-sec weighted for the neutron energy distr.)

$\Theta_{Tot} \rightarrow$ integral neutron flux $\int \emptyset(E)dE$

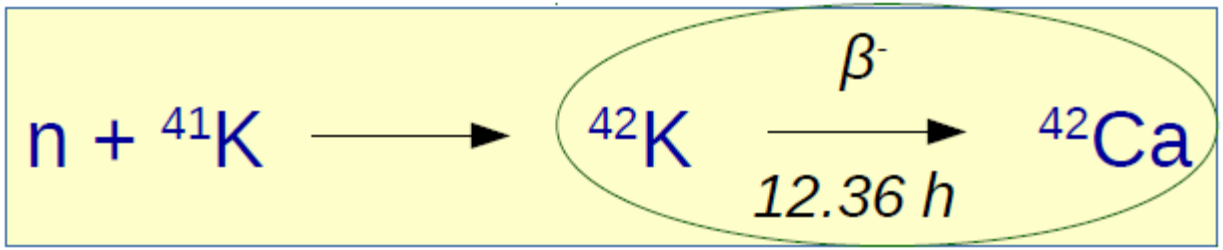
Sensitivity depends on:

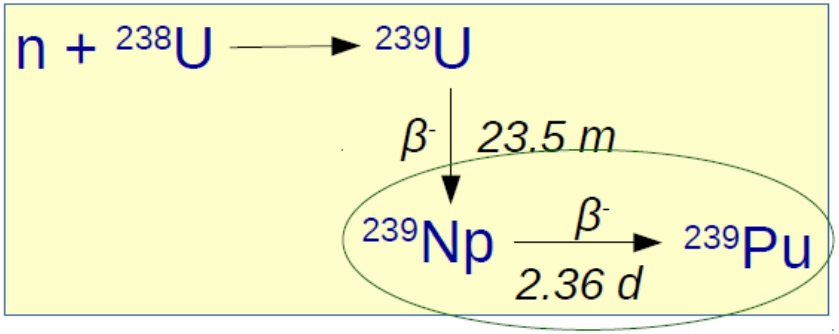
- type of material (short-lived activation products)
- neutron exposure time
- interferences in the matrix
- background in the region of the gamma emission

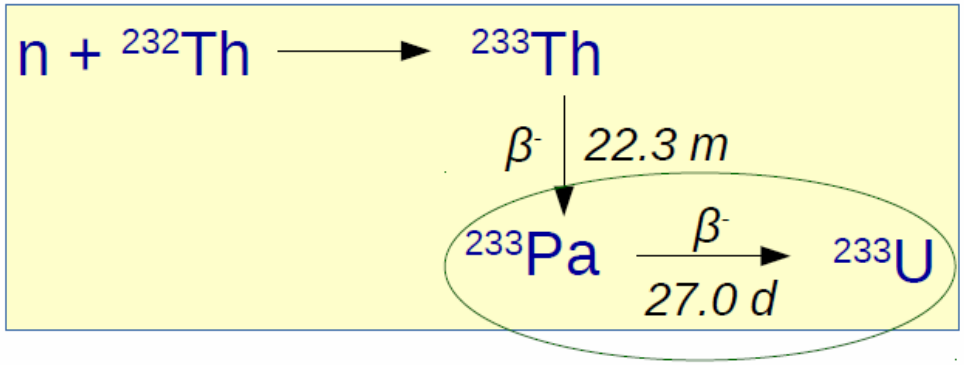
- For ^{40}K, NAA reaches far greater sensitivities than all other techniques

- ^{41}K isotopic abundance is 6.7%
- ^{40}K isotopic abundance is 0.01%

^{40}K contamination is calculated from ^{41}K one



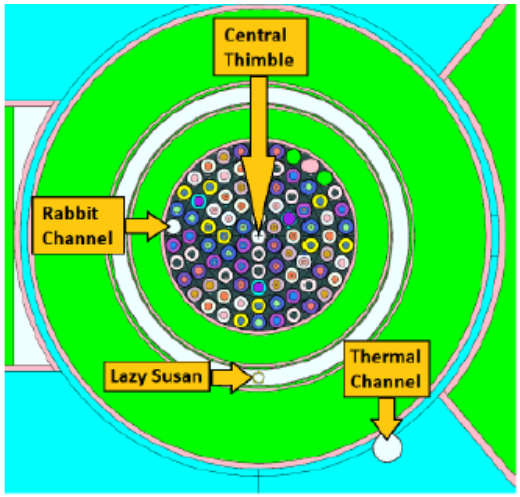




The material of the sample container should not form long-lived radioisotopes during neutron irradiation: too long cooling times after the irradiation may prevent measuring shorter living nuclides, like ^{42}K.

Neutron irradiation:

TRIGA Mark II
research reactor
(250 kW) - Pavia, Italy



Sample preparation and HPGe measurements at Milano-Bicocca

^{238}U → 0.012 mBq/kg – 1 ppt

^{232}Th → 0.004 mBq/kg – 1 ppt

^{40}K → 0.27 mBq/kg – 1 ppt

Sensitivity achieved on **Acrylic** @ INFN Milano-Bicocca
PRELIMINARY RESULT

	^{40}K [1E-12 g/g]	^{238}U [1E-12 g/g]	^{232}Th [1E-12 g/g]
Acrylic sample (8.2 g)	< 0.016	< 0.3	< 0.5

@ 90% C.L.

M. Sisti talk LRT-2019, Jaca

	^{40}K [1E-12 g/g]	^{238}U [1E-12 g/g]	^{232}Th [1E-12 g/g]	Ref.
SNO Acrylic	-	< 1.1	< 1.1	[1]
Borexino Liquid Scintillator	< 6.1	< 1.0 E-5	< 1.8 E-4	[2]
KamLAND Liquid Scintillator	< 2.4 E-3	< 1.0 E-5	< 5.5 E-3	[3]
EXO Heat Transfer Fluid HFE-7000	< 580	< 7.3	< 3.7	[4]
EXO Heat Transfer Fluid HFE-7000	-	< 0.015	< 0.015	[4]
EXO DuPont Teflon TE 6472 raw	1800±200	< 0.78	< 0.26	[4]
EXO APT Teflon	2010±200	< 1.2	< 0.62	[5]
MAJORANA Teflon TE 6472	150±20	0.025±0.002	< 0.4	[6]

[1] J. Boger et al., Nucl. Instr. and Meth. A 449 (2000) 172
[2] R.v. Hentig et al., Nucl. Phys. B (Proc. Suppl.) 78 (1999) 115
[3] Z. Djurcic et al., Nucl. Instr. and Meth. A 507 (2003) 680
[4] D.S. Leonard et al., Nucl. Instr. and Meth. A 591 (2008) 490
[5] D.S. Leonard et al., Nucl. Instr. and Meth. A 871 (2017) 169
[6] N. Abgrall et al., Nucl. Instr. and Meth. A 828 (2016) 22

NAA: Figure of Merit

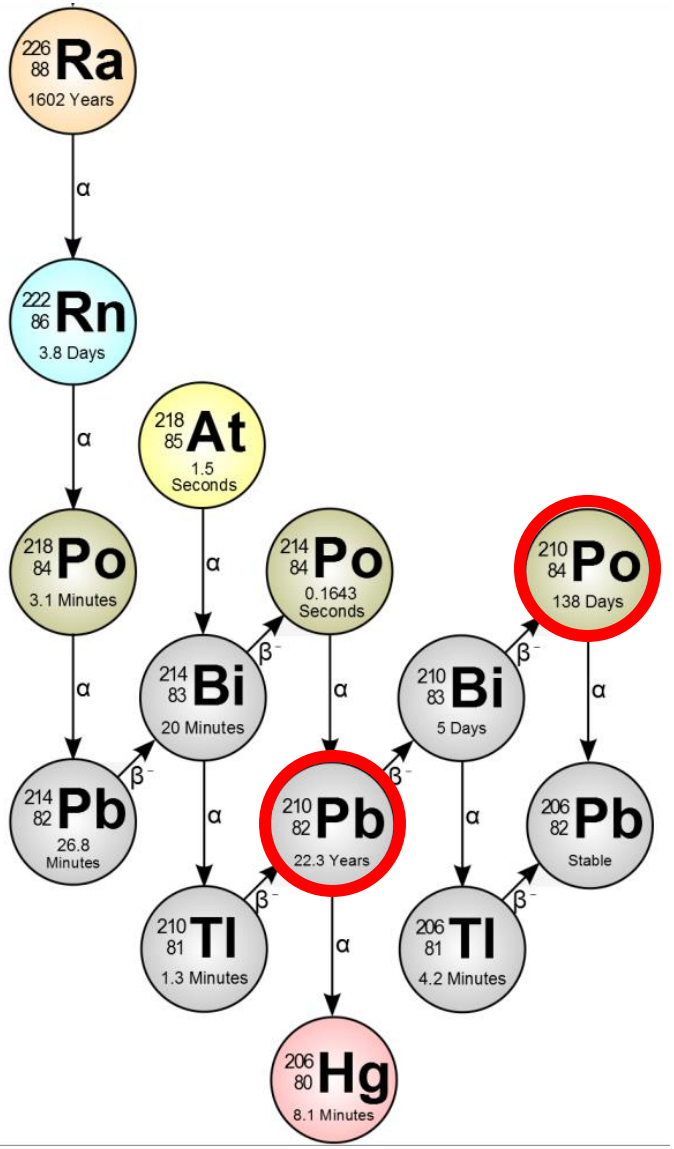
Advantages:

- **NAA can achieve substantially greater sensitivity than direct γ-ray counting**
- **For ^{40}K, NAA reaches far greater sensitivities than all other techniques**

Limitations:

- Trace elements analysis requires careful preparation of the irradiation campaign and of the test samples in order to reach sub-ppt sensitivities
- It is necessary to use irradiation standards (containing the same elements to be traced in the sample with a known amount)
- It cannot measure the daughters in the chain (no info on the equilibrium)
- You need to access to a nuclear reactor

Radio-chemical methods (^{210}Po extraction)



Background component:

- Surface contamination
- Bulk contamination
- (α,n) reaction

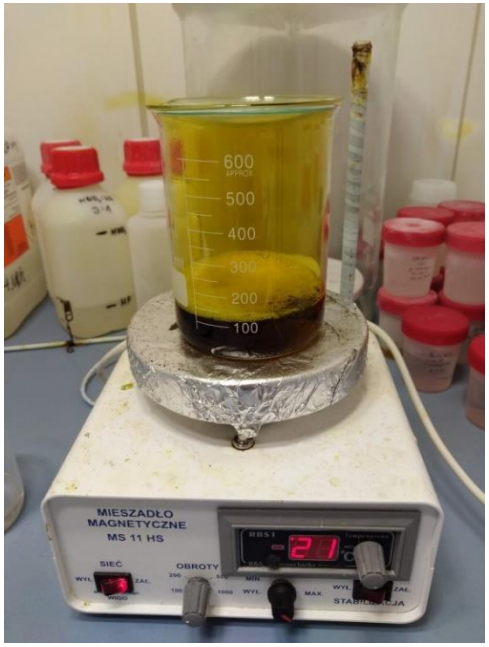
Radioanalytical/Radiochemical problems:

- measurement can be done only by alpha spec
- High volatility of Po – only wet ashing in boiling concentrated acids and acid mixtures (problems with some polymers materials)

Chemical separation of ^{210}Po

Separation method of ^{210}Po:

- Sample etch and dissolution
- Sample pre-concentration and purification (if needed) by co-precipitaion or ion-exchange chromatography (e.g. measurement of bulk ^{210}Po in Cu samples)
- Source preparation by spontaneous deposition



Radiochemical tracers:
^{208}Po (5.115 MeV alphas)
^{209}Po (4.883 MeV alphas)

Reagents:

- HNO_3
- HCl
- H_2O_2 30%
- $N_2H_4 \bullet 2HCl$
- Silver discs

Laboratory equipment:

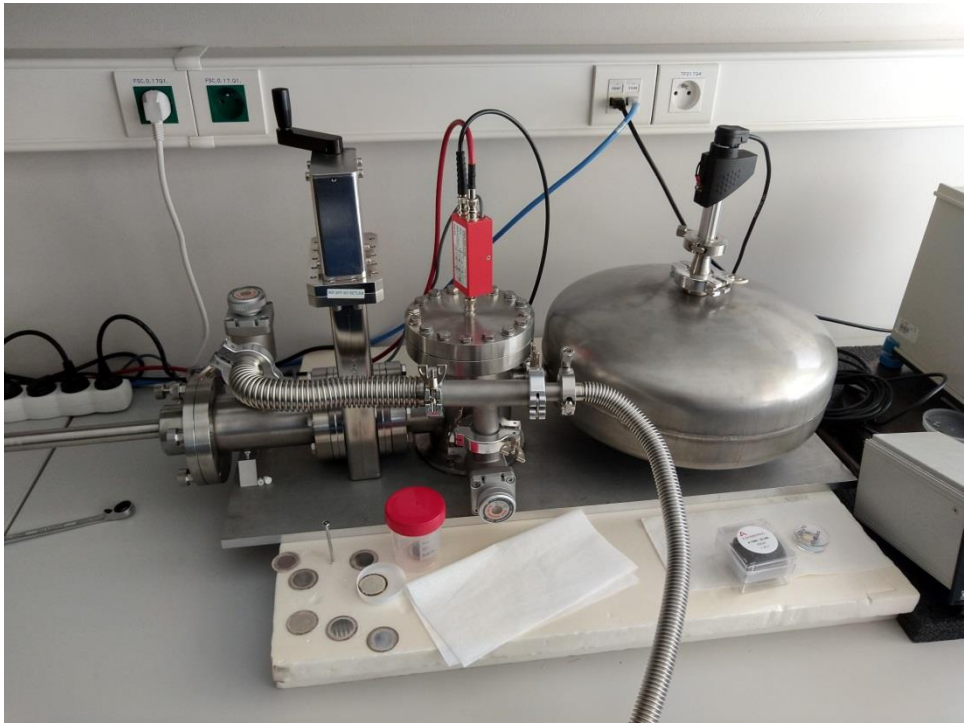
- Glass beakers (250 ml)
- Watch glasses
- Stirring rods with PTFE coating
- Hotplate with stirrer
- Pipette 10-100 µl+ tips
- Protective equipment (glasses, gloves etc.)

Courtesy of T. Mróz
Jagiellonian University in Cracow, Poland

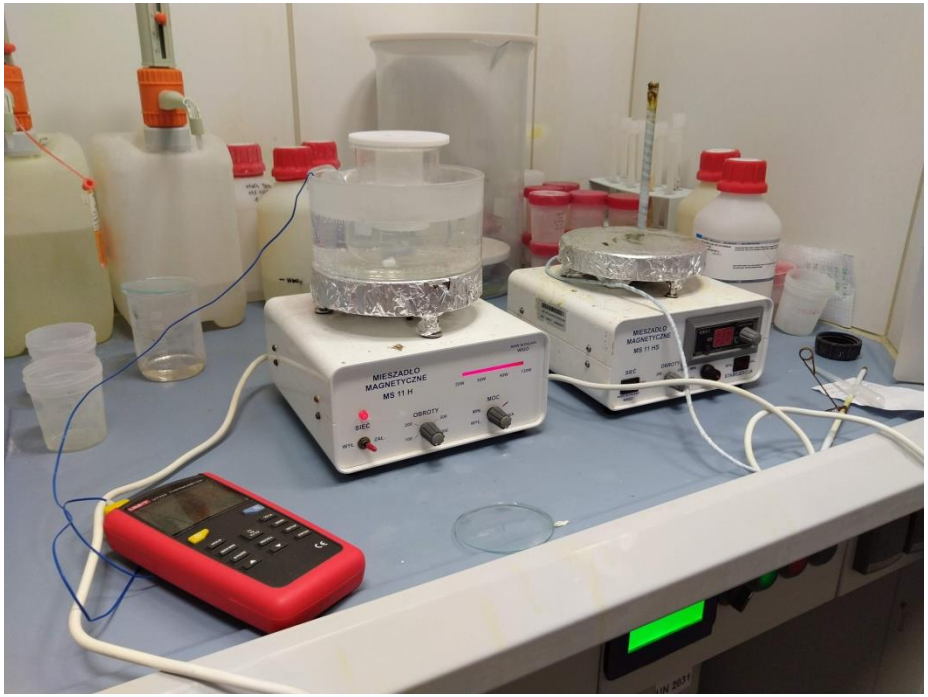
Detection of ^{210}Po – semiconductor alpha spectrometer

- Very good energy resolution (20-30 keV FWHM)
- Detection limits for activity lower than for XiA

- Requires radiochemical separation of ^{210}Po
- Efficiency determined with tracer
- Spectrum resolution depends on the source quality
- Blank runs required to control the background for each procedure



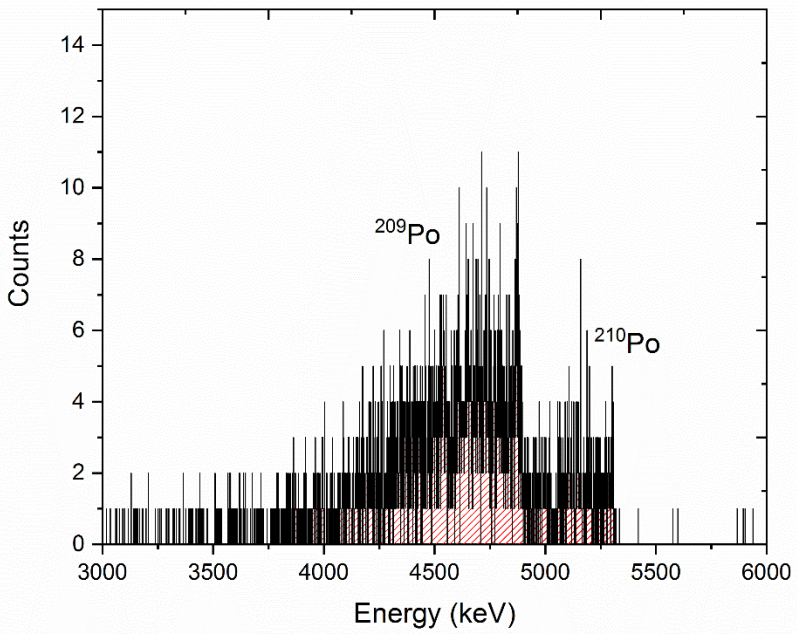
Source preparation



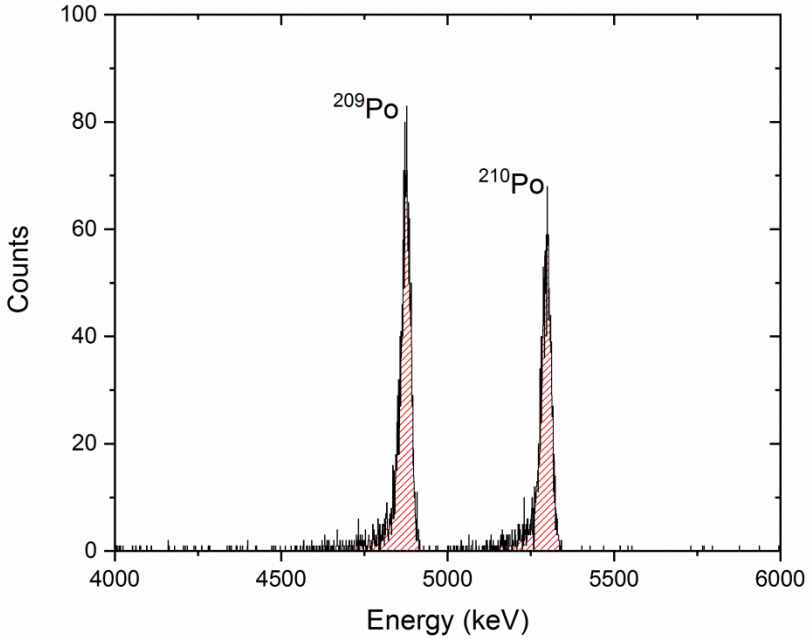
Spontaneous deposition of Po on Ag from diluted HCl solution

Alpha spectrometry sources

Thick layer deposited on source



Thin source (~20 keV FWHM)



Radio-chemical methods : Figure of Merit

Advantages:

- **Is the only technique able to measure the lower chain**

Limitations:

- Sample preparation

OBJECTIVES of this lecture

After this class, the student should be able to:

- Know the most important sources of **natural radioactivity.**
- Understand the principles of secular **equilibrium and disequilibrium** of the decay chains.
- Grasp the principles of the **cosmogenic** production of radionuclides.
- Understand the general framework of the problems posed by the **Rn-222.**
- Figure out the **importance of material assays and radiopurity** related aspects for low-background techniques.
- Know the principle of the **most used techniques** for the assays.

Thank you for your kind attention!

Backup

ORGANIC ELEMENTAL ANALYSIS

Organic elemental analyzers determine Carbon, Hidrogen, Sulfur and Nitrogen content in a variety of materials.

Leco CS-244
METAL SAMPLES

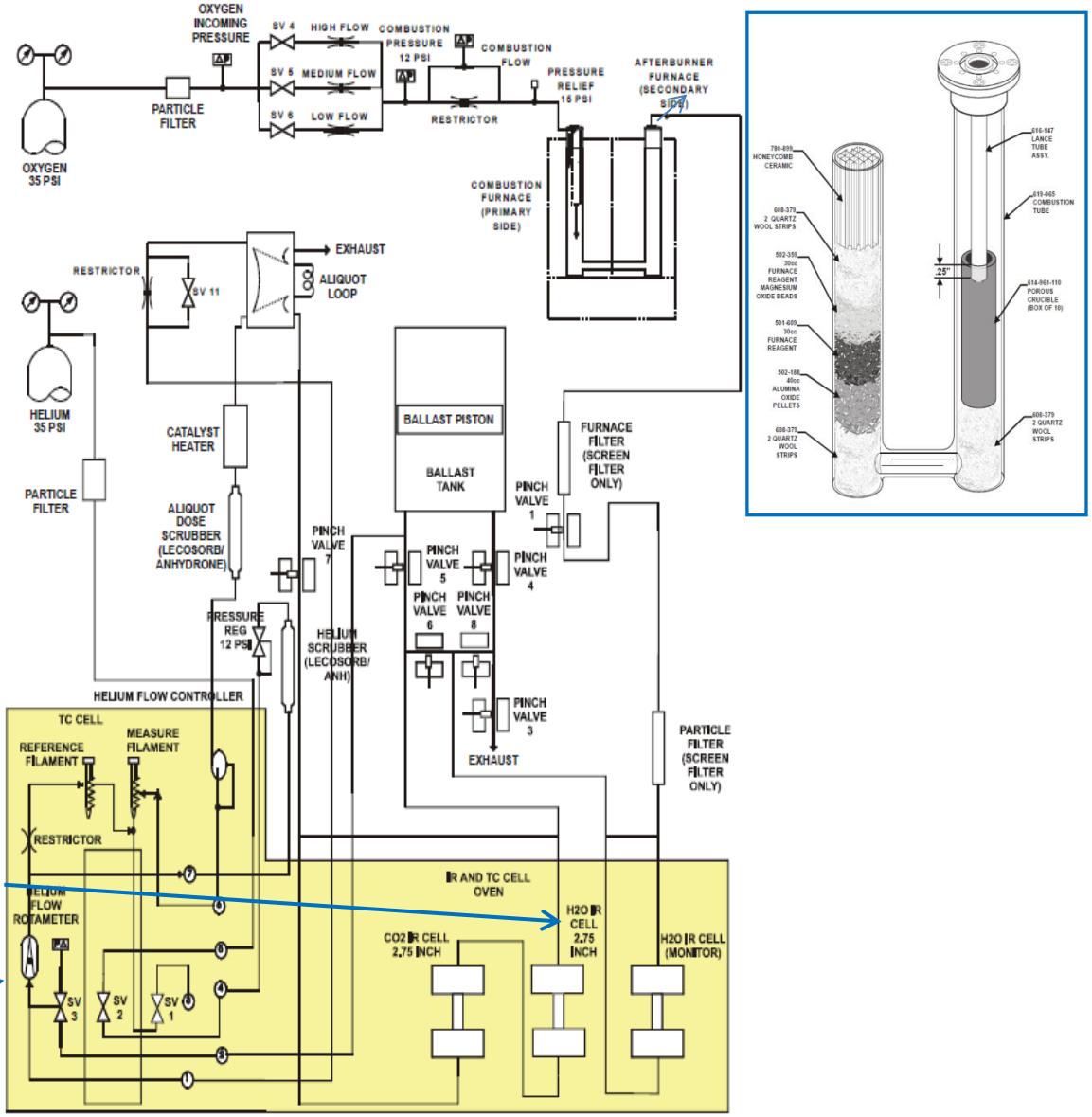
Leco TruSpec S detector
ORGANIC SAMPLES

There are 3 fases during analysis cycle: PURGE, COMBUST AND ANALYZE

- PURGE: The encapsulated sample is placed in the loading head, and purged of any at mospheric gases

- COMBUST: The sample is dropped into a hot furnace (950ºC) and flushed with oxygen for a complete combustion. The products of combustion are then collected in a collection vessel known as ballast.

- ANALYZE: The combustion gases are purged through the CO2 and H2O infrared detectors to analyze carbon and hidrogen . A thermal conductivity cell is used to determine the nitrogen content. Analysis of S requires an extra module



Elemental analysis of polymeric sheets

Reference		%C	%N	%H
1- DARK GADM	FSB-HFT	13,4	0,94	1,5
2 - DARK GADM	TRIPLEX-FSB	12,6	0,17	1,2
3- DARK GADM	RSD-HRT	13,3	1,7	1,9
4- DARK GADM	Cubo de fibra	59,3	5,9	5,3

CONCENTRATION RANGE % BY WEIGHT