Primary radioactivity metrology

2 November 2022

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SPARTE
Scintillating Porous Architectures for RadioacTivE gas detection

This project has received funding from the European Union's Horizon 2020 research and innovation programme under grant agreement No 899293

"Science of measurement associated with the evaluation of its uncertainty" (Academy of Sciences)

- **A measurement result has three elements:**
  - the numerical value obtained:

    $$3$$

  - the unit of measurement (chosen in the International System): the value of the physical quantity is the product of the numerical value by the unit used:

    $$L = 3\ \text{m};\ L = 3 * 1\ \text{m}$$

  - the third parameter quantifies the doubt on the measurement: it is the uncertainty

    $$L = (3 \pm 1)\ \text{m}$$

Unit is important:

- Mars Climate Orbiter (1999)
  - Two development teams using different unit systems:
  - Imperial unit: pound-force
  - SI unit: newton
- Scale error: One pound-force is equivalent to 4.48 newtons

  ➔Destruction of the NASA probe, Cost: US$ 125 M

- 1983, Air Canada 143 :
  - Confusion litres/gallon, Underestimation of the amount of fuel needed (factor 1.77), Fuel failure at 12,000 m, half way through the trip.

  ➔ Emergency landing after gliding... no victims.

- 1999, cargo Korean Air:
  - Altitude confusion at 1500 meters: control tower in meters and pilot in feet...

  ➔ 8 dead, 37 injured

Uncertainty is important

- **Assigning a value to the uncertainty allows:**
  - to quantify the quality of the measurement result,
  - to verify that results can be in conformity with a physical law,
  - to decide on the compatibility of several results,
  - to have sufficient information for decision making, ...
- **Important (for managers): the evaluation of the uncertainty makes it possible to adapt as accurately as possible (neither too much nor too little) the means implemented (equipment, personnel) to achieve the desired objective**

Evolution of metrology in the word and in France

Standard: the king

Signature of the Metre Convention
Creation of the BIPM

BIPM

1ère CGPM

Creation of the International System of Units (SI)
11ème CGPM

BNM creation in France

IN France BNM become LNE
LNHB from CEA obtained the mission of activity and dose metrology

The last physical standard (kg) is replaced with Keeble balance, all the SI units are connected to a physical constante

1791-1795 | 1875 | 1889 | 1946 | 1954 | 1960 | 1961 | 1969 | 1971 | 1975 | 2005 | 2018

1er août 1793
National Convention
Adopts the metre

3 units:
m, kg, s

MKSA

K,
candela

Ci, Gy
and Kerma

mole

Bq and Gy

International organisation

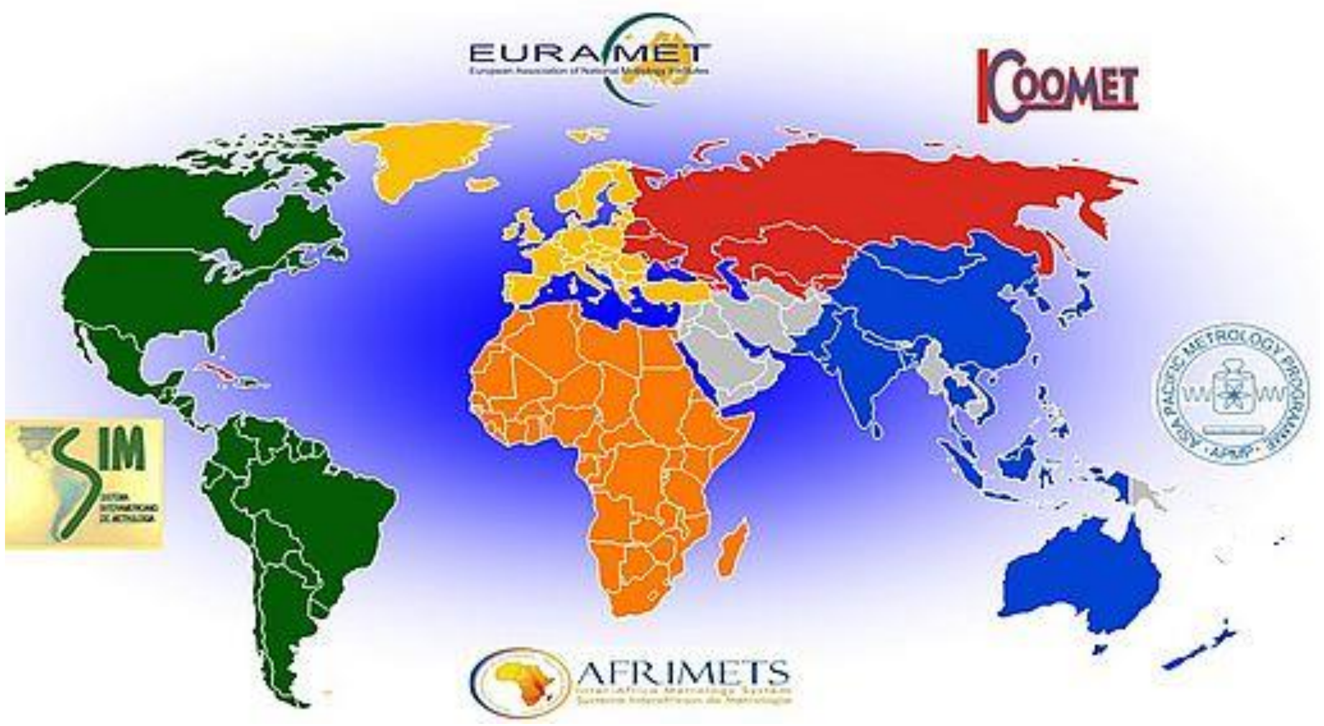
- Bureau International des Poids et Mesures (BIPM) - Pavillon de Breteuil in Sèvres

- "to ensure worldwide uniformity of measurements and their traceability to the international system

~~Altruism, desire to reach scientific perfection?~~

- "...the lack of uniformity of units is a source of error and facilitates fraud in commercial transactions. "

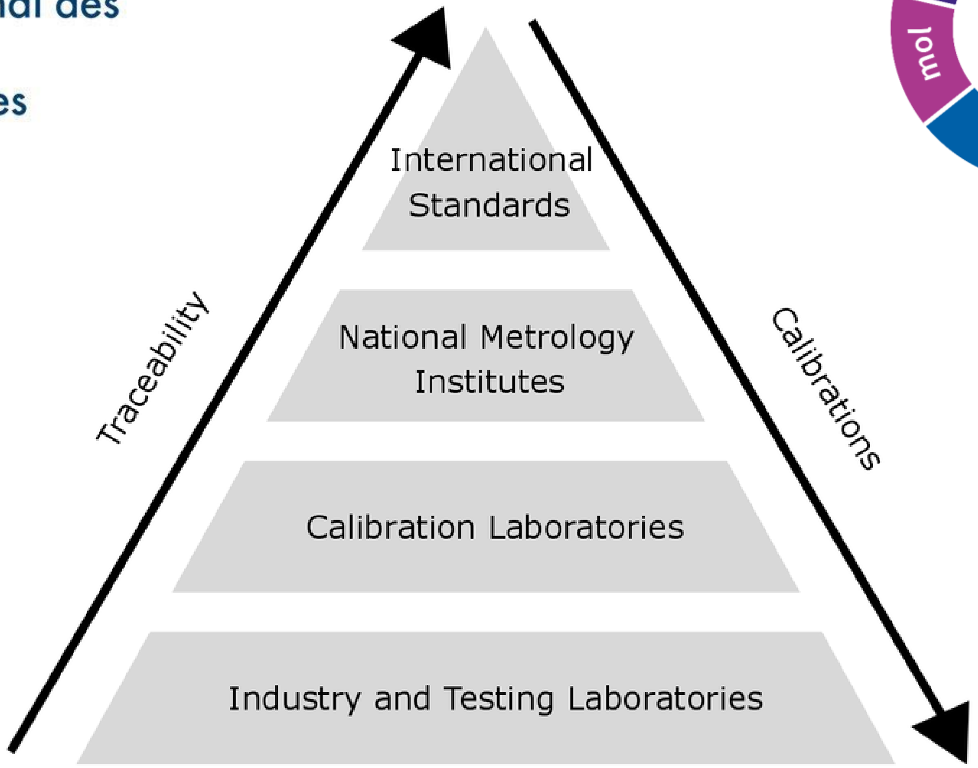
Advisory committees (depending on the field), regional organizations (EURAMET in Europe)



The BIPM ensures, together with the National Metrology Institutes (NMIs), the equivalence and mutual recognition of calibrations traceable to the International System (SI) throughout the world.

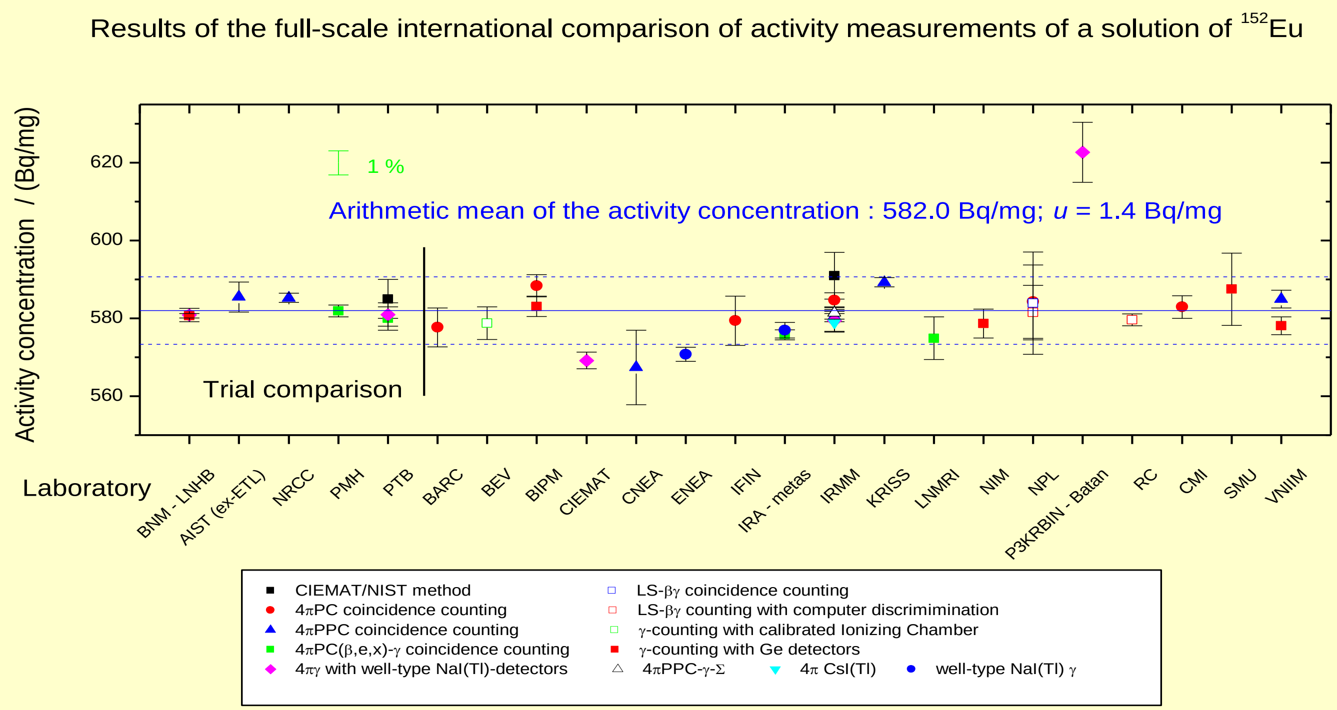
Metrological traceability chain: succession of standards and calibrations that is used to link a measurement result to a reference

Bureau International des Poids et Mesures



Metrology for radionuclides

- Activity : average number of disintegrations per time unit
- Unit : becquerel (Bq = 1 s^{-1})
- Primary measurement methods : developed by national metrology institutes (NMI)
- International comparison between NMI performed at BIPM (when it is possible)



Primary measurement methods, examples in activity metrology

Two types of measurement methods

Relative Method

$$A = A_0 \times \frac{S}{S_0}$$

- A: unknown activity of source S
- A_0: activity of the standard S_0
- S: signal due to source S
- S_0: signal due to the standard

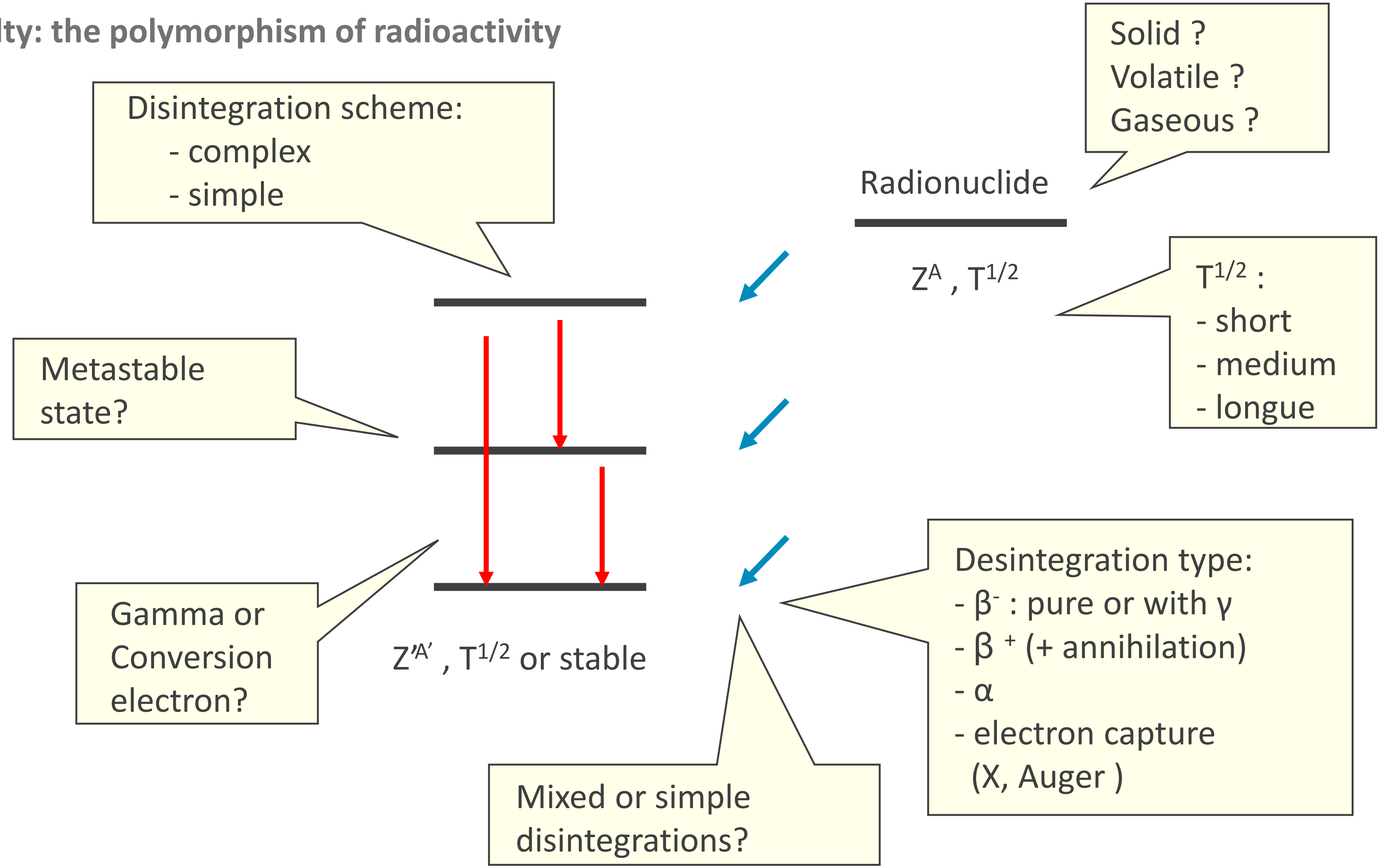
Primary Method

$$A_0 = \frac{S}{R}$$

- R: overall source/detector efficiency
- R is a priori unknown, it can be: measured, calculated, extrapolated to 1
- Other possibility: formula independent of R

Measurement methods

Difficulty: the polymorphism of radioactivity



Measurement methods

In general

- **The variety of emitted radiation and the physical forms of the sources make it necessary to adapt the measurement procedures to each case in order to establish the national references:**
    - defined geometry method, in which the detector is exposed to a limited and known fraction of the radiation,
    - 4π geometry methods where the detector is exposed to the totality of the radiation,
    - coincidence method, applicable to radionuclides emitting simultaneously at least two types of radiation (β-γ, α-γ...), ...

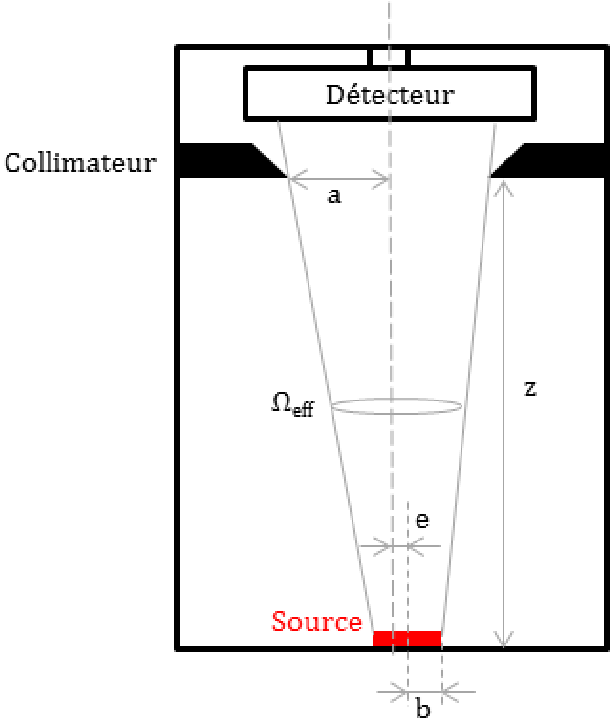
- **The "ideal" primary method must be accurate, precise, under statistical control*, independent of decay scheme parameters and independent of other radioactivity standards**

- *** The fluctuations observed during the measurement repetitions are compatible with the known sources of uncertainty**

Defined solid angle method

Defined solid angle method

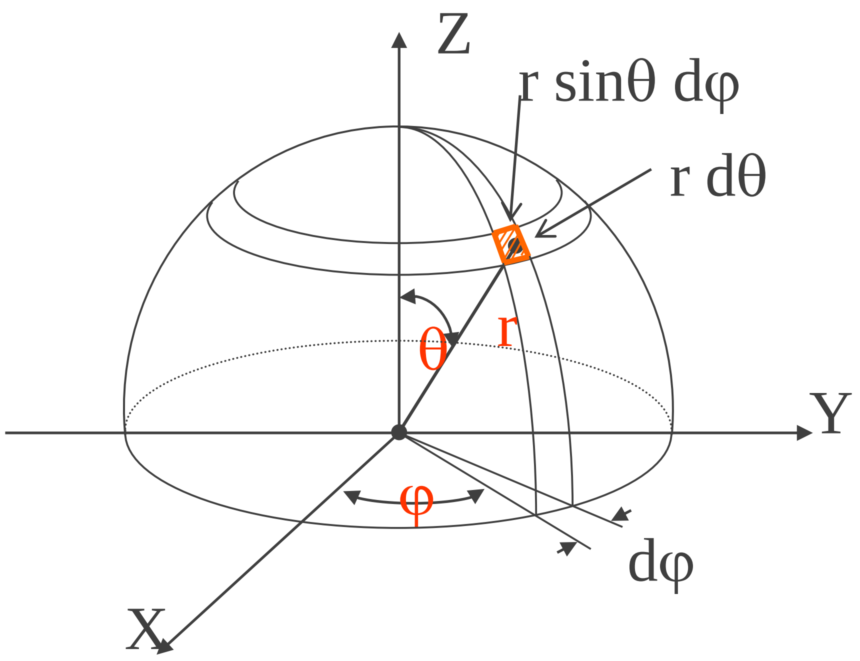
- **Assumptions: a particle emitted by decay propagates in a straight line in a random direction, each particle entering the detector is counted, the geometry is well defined**
- **Efficiency :**
  - detector = 1
  - geometric = solid angle / 4π sr
- **Simplifications :**
  - axial symmetry,
  - point or circular source,
  - circular diaphragm,
  - parallel planes,
  - homogeneous activity distribution in the source



- **In reality : inhomogeneous source, non-circular and off-axis the edges of the diaphragm have a non-zero thickness**

Defined solid angle method

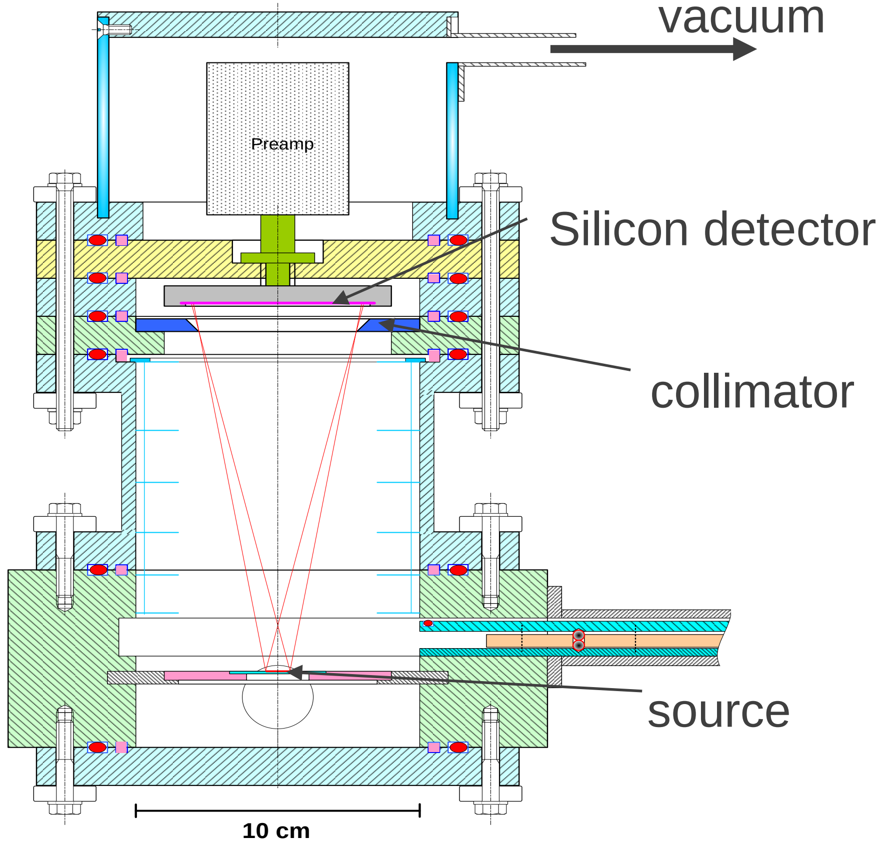
Alpha counter with defined geometry: how to define it?



$$ds = r^2 \sin\theta \, d\varphi \, d\theta$$

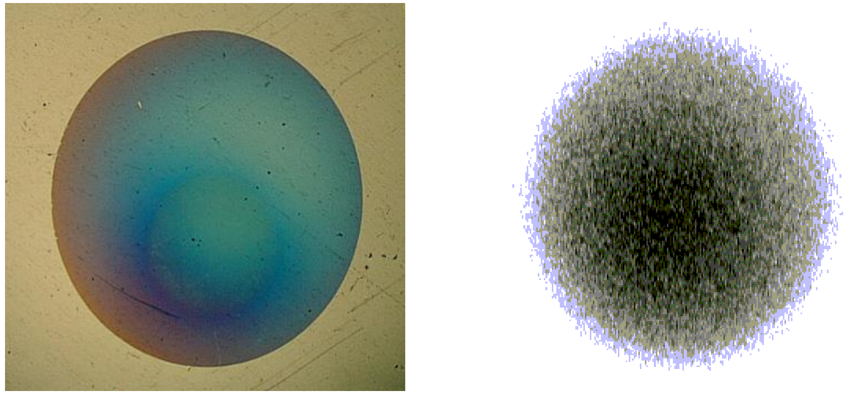
$$d\Omega = \sin\theta \, d\varphi \, d\theta$$

$$\Omega = s/r^2$$

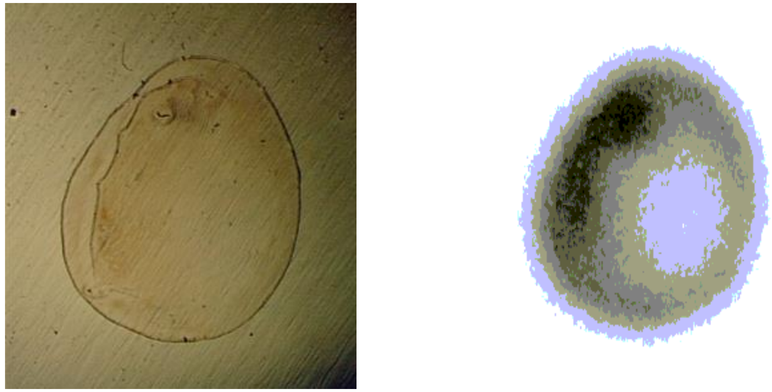


Defined solid angle method

- **For which radiation?**
  - The radiation must not be scattered in the measuring chamber or by the collimator and the detection efficiency of the detector must be known.
- **Method usable for: alpha particles (MeV), X-rays < 10 keV**
- **With what type of source?**
  - Small disks (a few mm in diameter) manufactured with different methods:



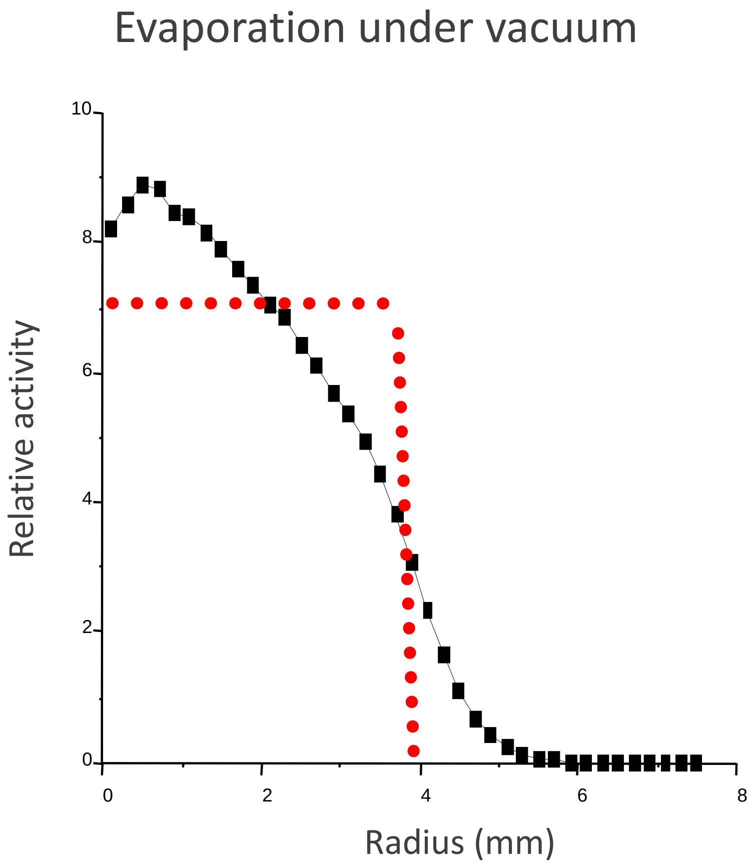
Evaporation under vacuum

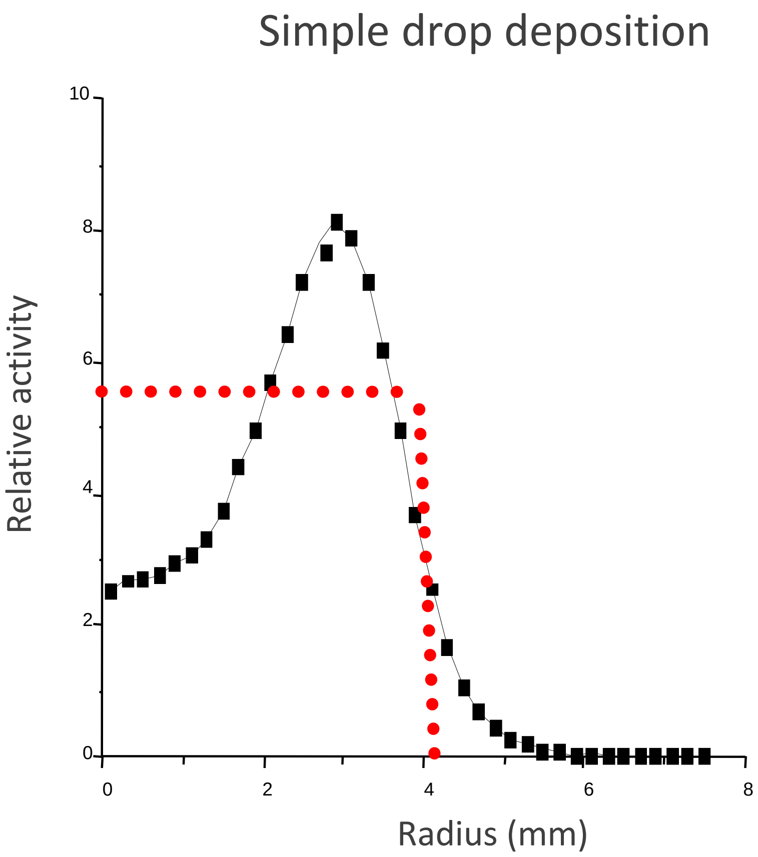


Simple drop deposition

Defined solid angle method

In practice, these sources are not homogeneous





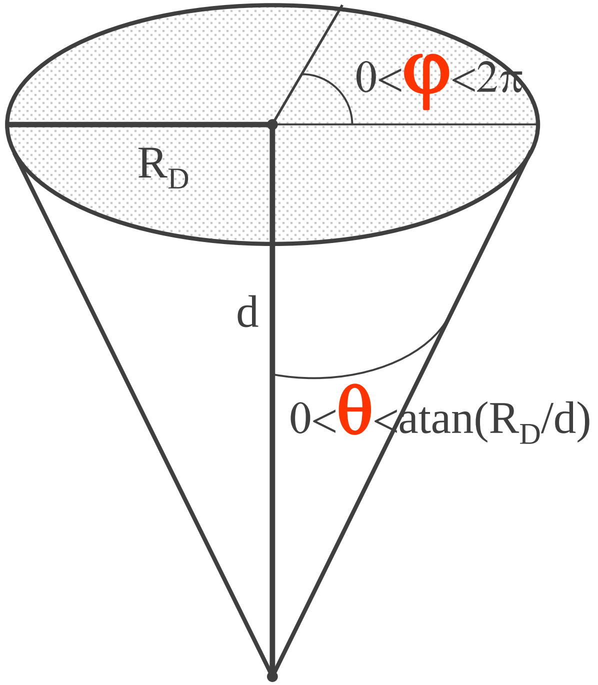
Defined solid angle method

- **Calculation of the solid angle: case of a point source**
  - Only geometry with a simple solution to calculate Ω

$$\boxed{\Omega = 2\pi(1-\cos\theta)}$$

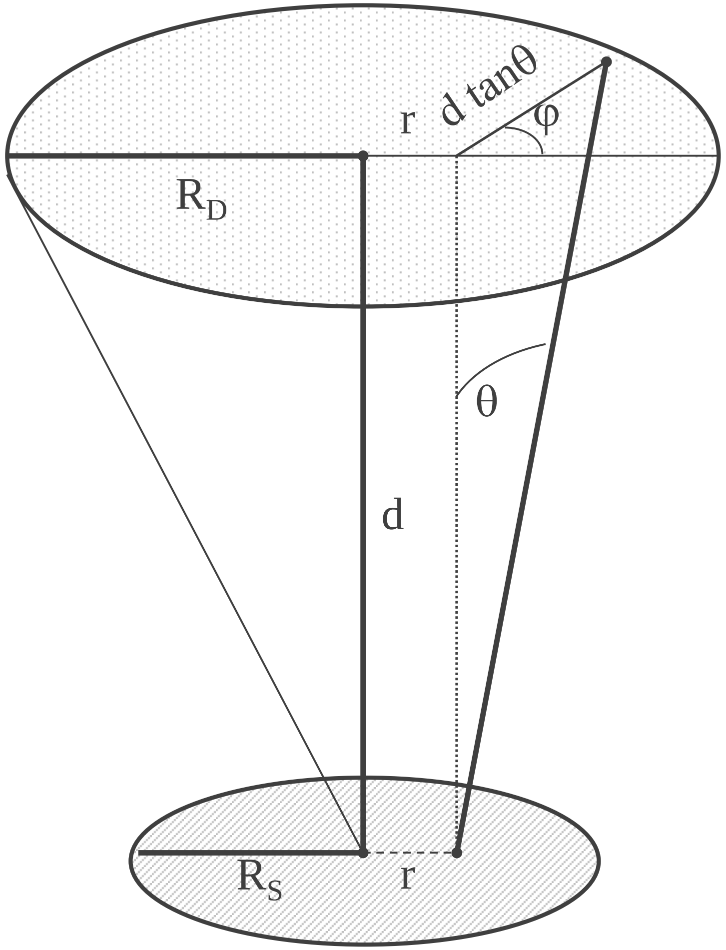
$$\theta = a\tan(R_D/d)$$

Circular diaphragm



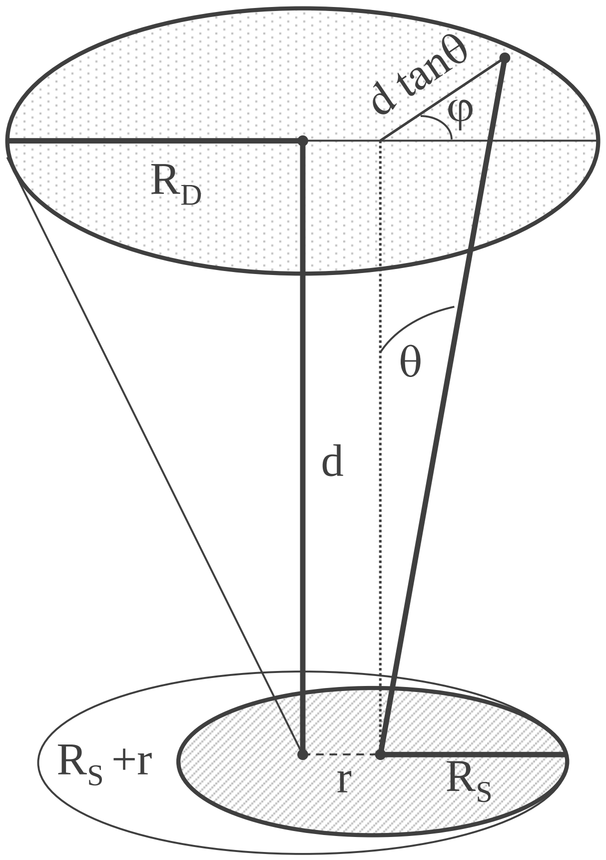
Point source in the axis

- **Calculation of the solid angle: case of a circular homogeneous and coaxial source**
  - Tables and approximations
    Masket, Jaffey, etc.
  - Numerical integration
    Pommé et al., NIM A505
  - Elliptic functions
    Tryka, Optics Com. 137
  - Bessel functions
    Ruby, NIM A337
    Pommé, NIM A531



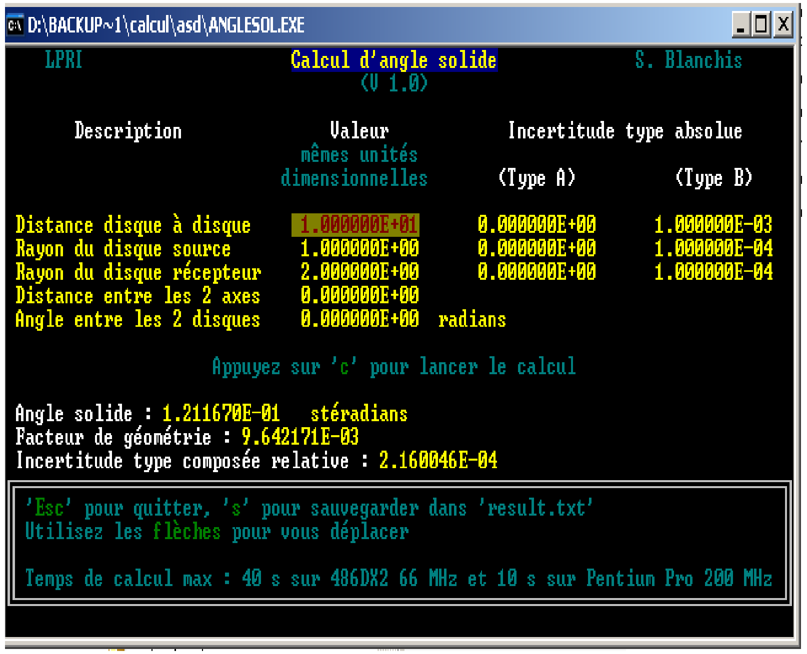
Calculation of the solid angle: Point source or circular homogeneous off axis

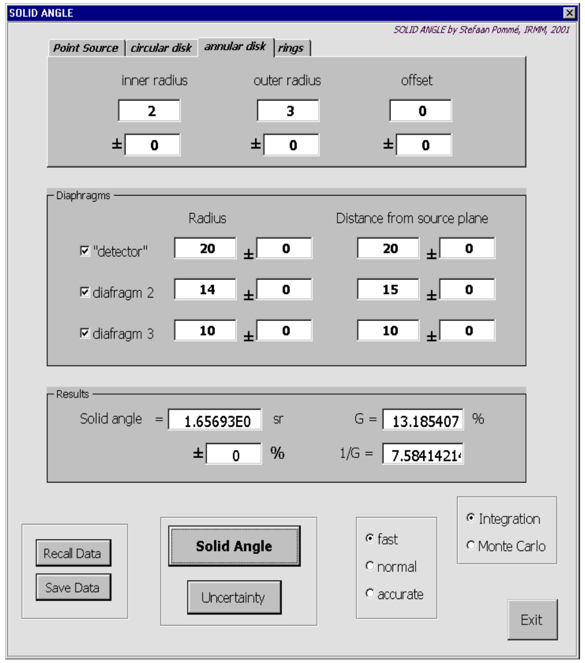
- Elliptic functions
  Tryka, Optics Com. 137
- Bessel functions
  Ruby, NIM A337
  Pommé, NIM A531



Defined solid angle method

- **For the calculations, small softwares exist:**
  - ANGLESOL (LNHB) S. Blanchis, Note technique LNHB 97/020. CEA - LNHB F- 91191 Gif-sur-Yvette Cedex
  - SOLIDANGLE (JRC, Belgium)





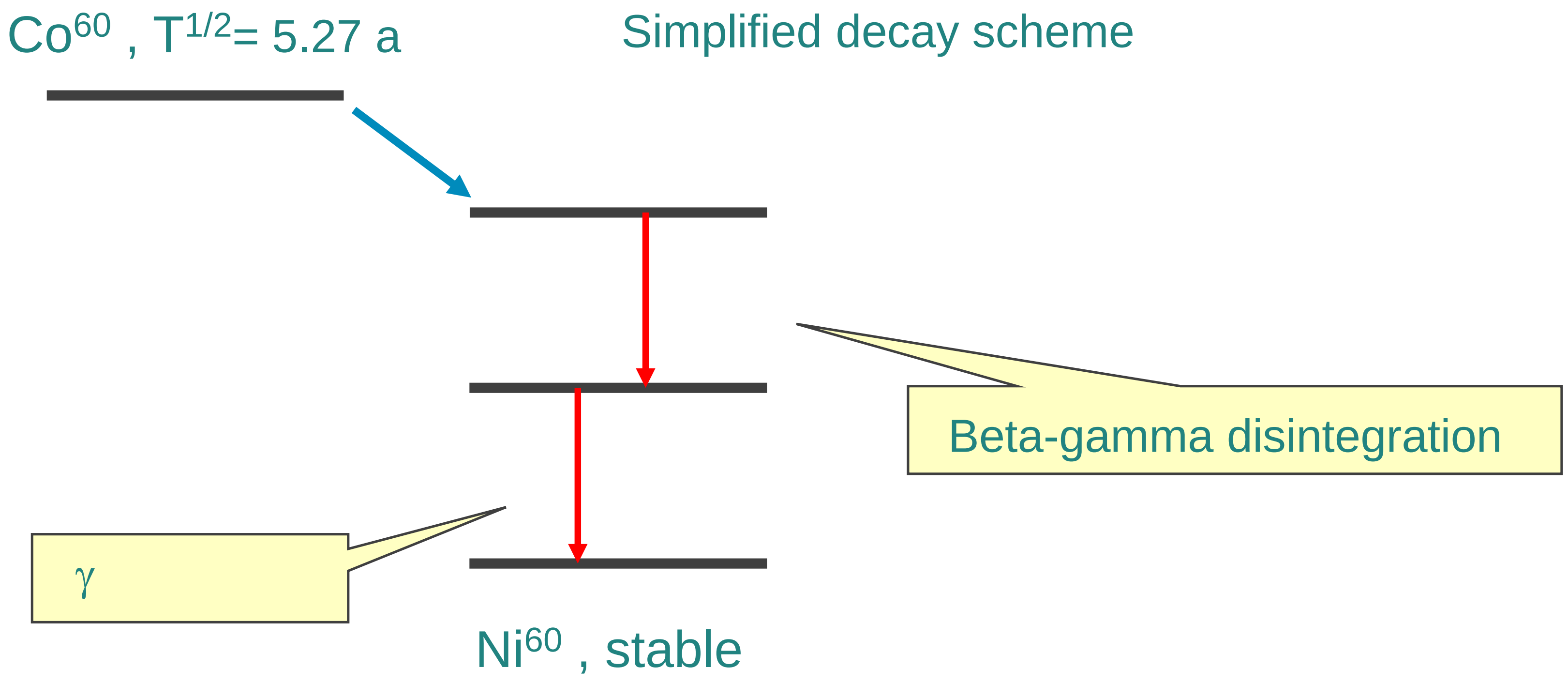
- **The solid angle method can be simple to set up as long as distances can be measured. The most complex part remains the manufacturing of the sources (very fine deposit required)**

Defined solid angle method

- **Some examples:**
  - For solid alpha sources:
  - For Rn-222 gas...

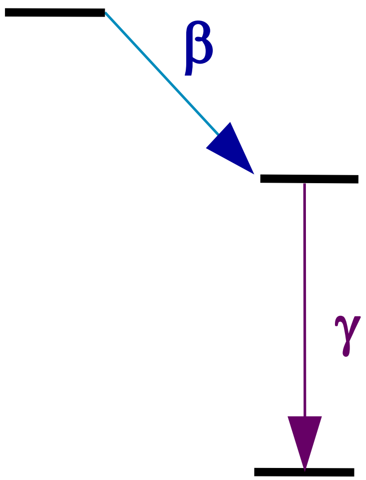
Coincidence measurement method

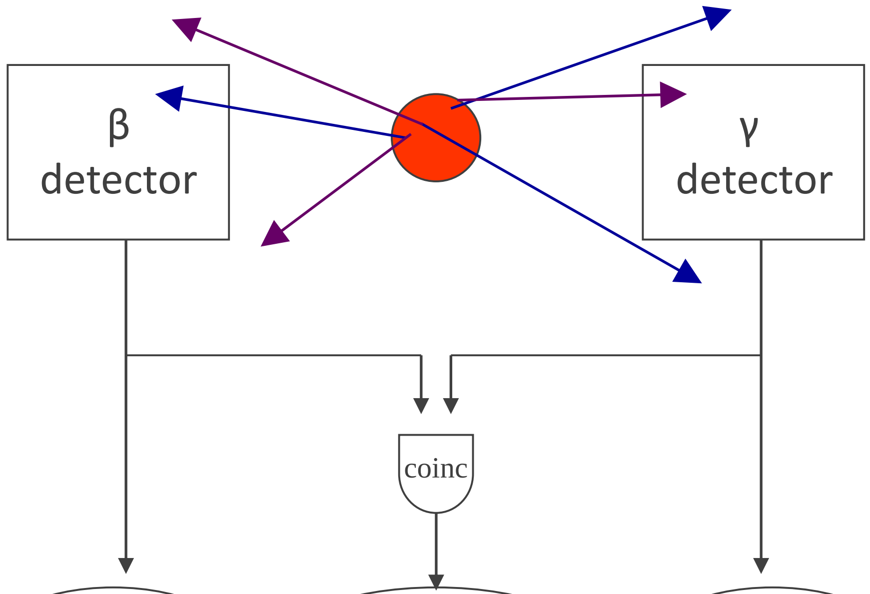
- **Example: ^{60}Co activity measurement**



Coincidence measurement method

- Principle of the method: Measurement of the count rate of each detector and the coincident count rate





$$N_\beta = A\,\varepsilon_\beta \qquad N_c = A\,\varepsilon_\beta\,\varepsilon_\gamma \qquad N_\gamma = A\,\varepsilon_\gamma$$

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

- Three equations... three unknowns but only under certain conditions

Coincidence measurement method

Conditions:

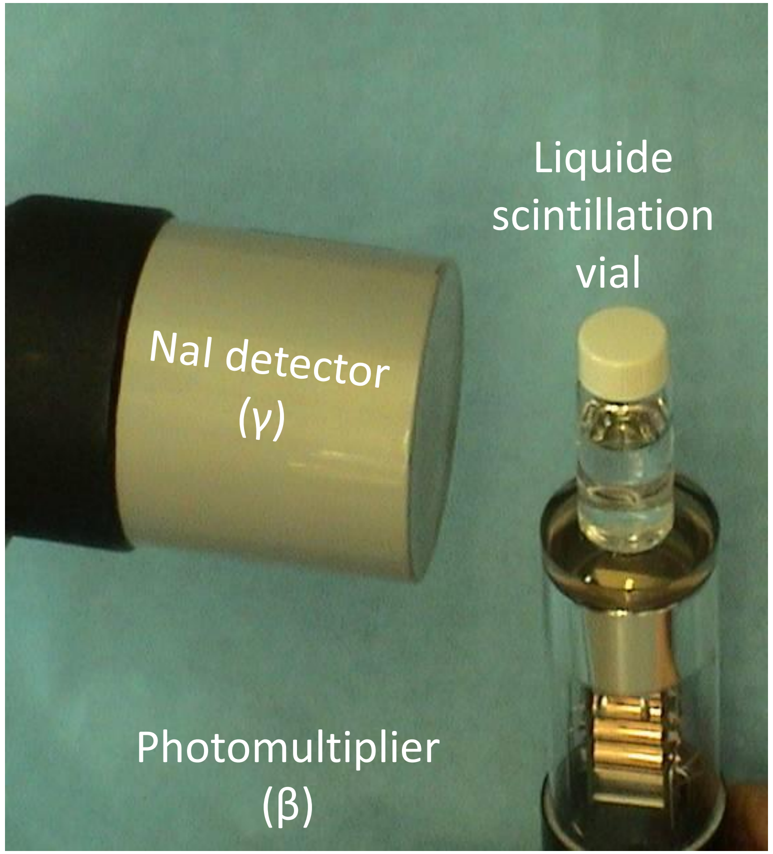
- **The detectors should only see one type of radiation**
  - No γ detection in the β detection
  - No noise in the counters!
- **ε_β and ε_γ must be independent and constant**
  - No angular correlation between β and γ use of a β 4π detector
  - At least one of the yields must not depend on the decay location in the source
- **No coincidences should be lost**
  - Coincidence window wide enough to compensate for jitter between β and γ signals
  - Correction of coincidences and dead time

Coincidence measurement method

Usable detectors :

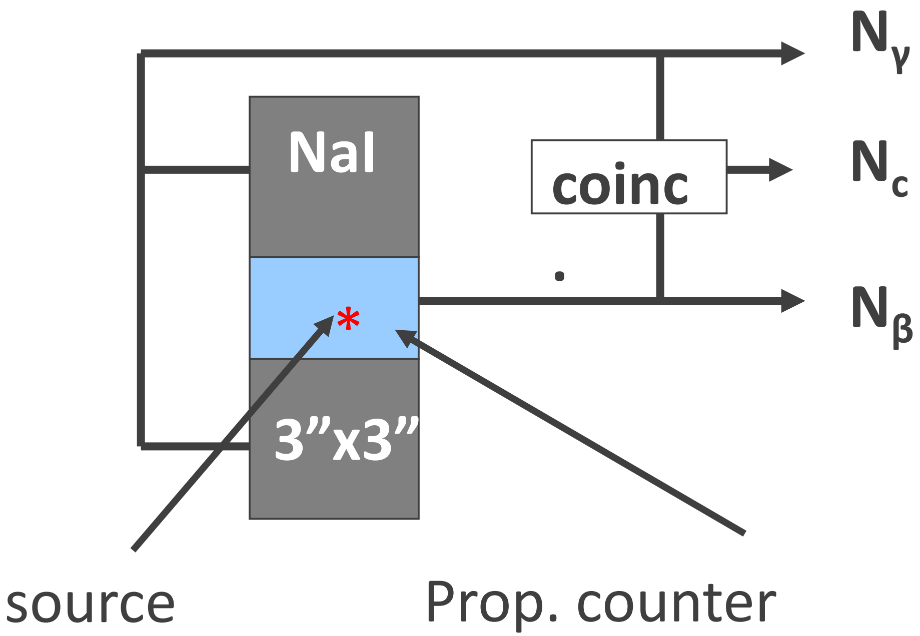
- **Geometry 4**
  - Good performance reduces extrapolation uncertainties
  - Suppresses angular correlation
  - Avoids scattering and absorption phenomena outside the source
- **Proportional counters and thin sources**
  - Good efficiency
  - Almost all particles leaving the source are counted
  - But need to chemically stabilize the solid source if the source is volatile (ex : iodine, antimony,...)
- **Liquid scintillation counters**
  - Easy to prepare sources
  - But higher g detection efficiency than proportional counters

- A rudimentary example...

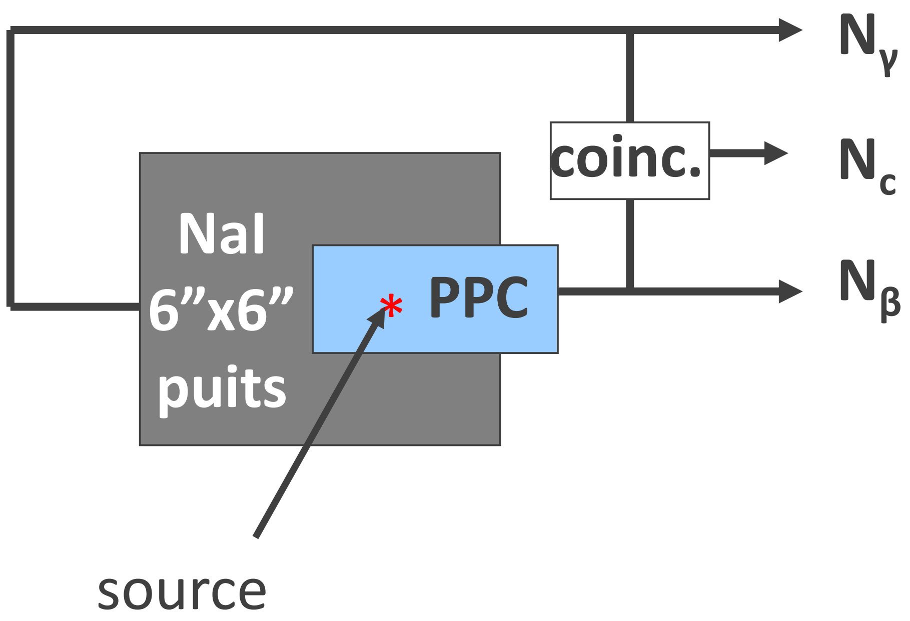


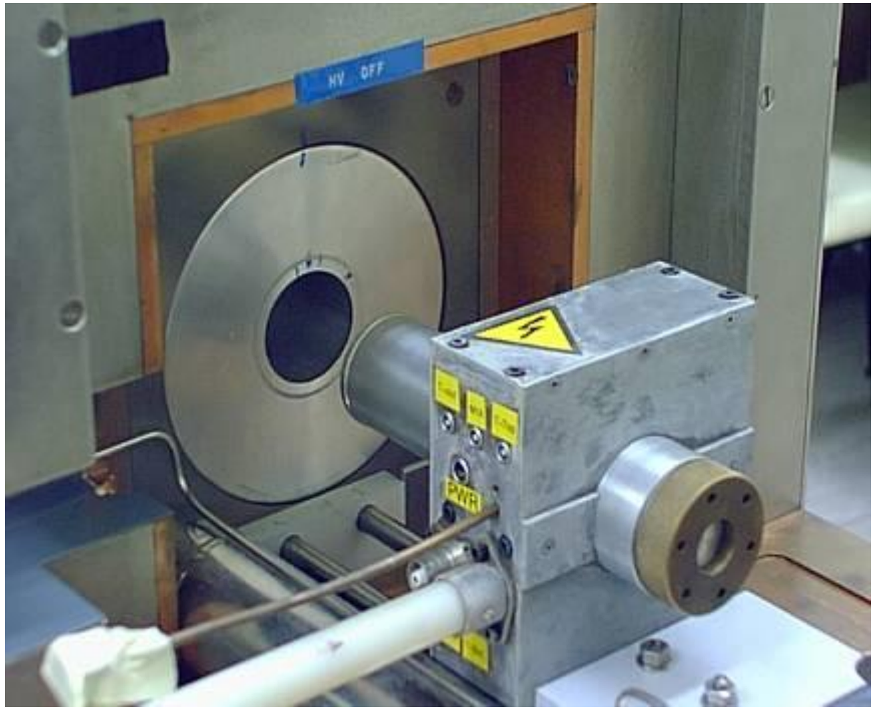
Coincidence measurement method

A classic 4π β-γ system operating at atmospheric pressure



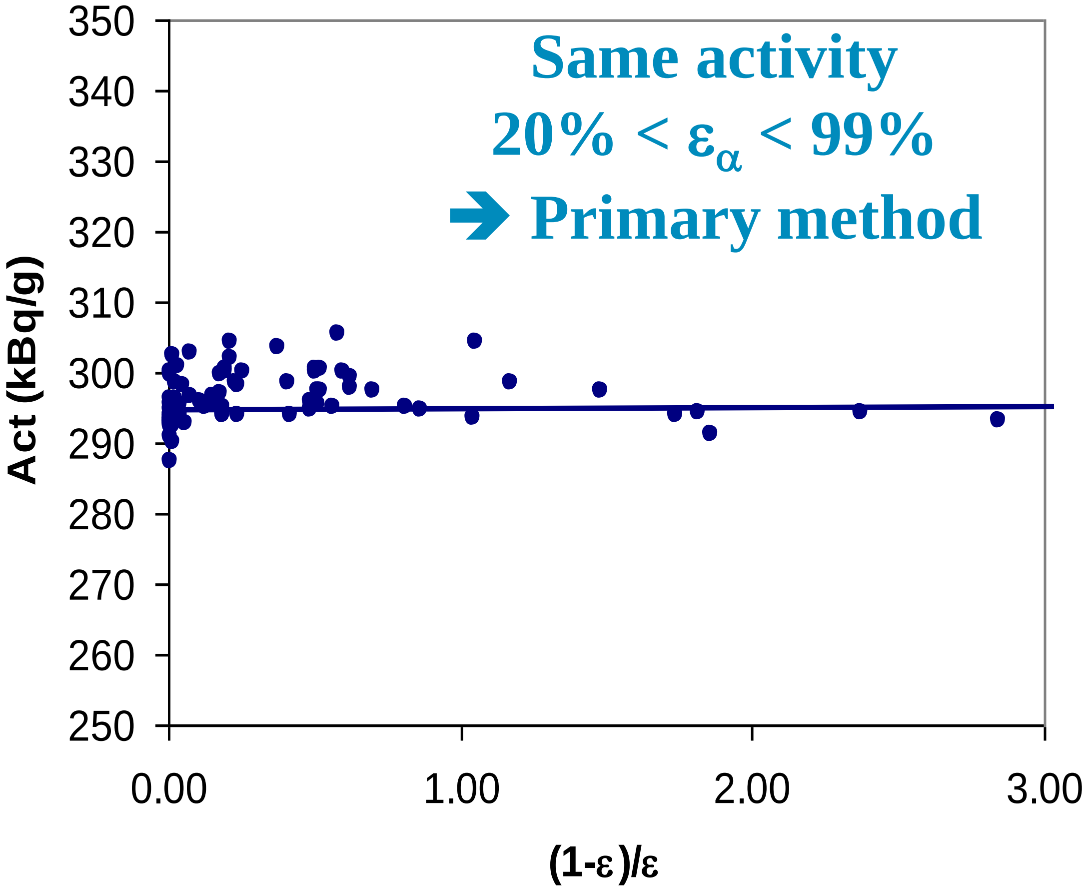
An efficient system

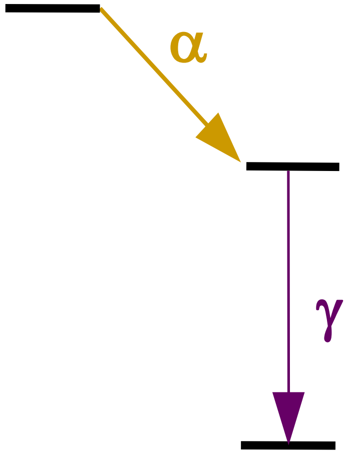




PPC = pressurised proportional counter

- Example: ^{241}Am by coincidences α–γ





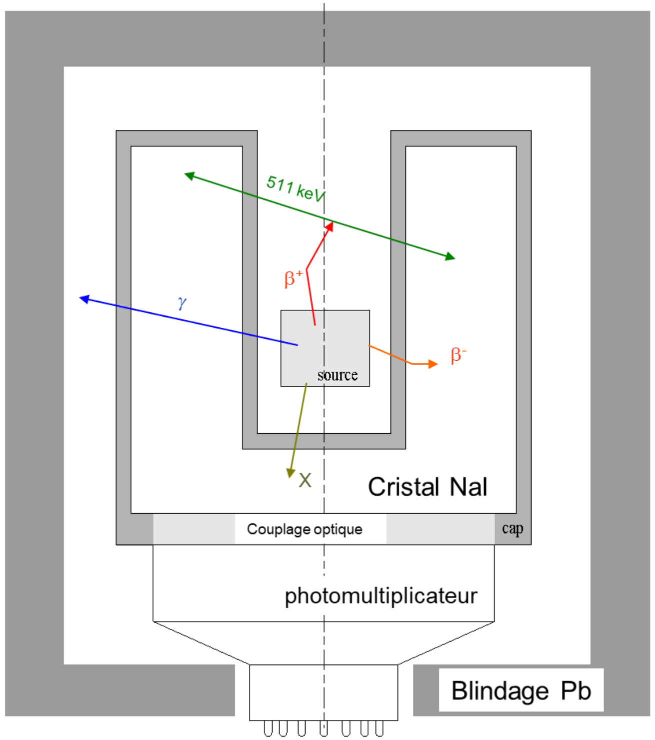
$$\varepsilon_\alpha = \frac{N_c}{N_\gamma}$$

Coincidence measurement method

- **Small additional problems...**
  - Non-uniform extended sources ?
  - Sensitivity of the beta detector to X or γ photons and conversion electrons ?
  - Sensitivity of the γ detector to annihilation photons in case of β+ decay ?
  - Complex multi-branch decay patterns ?
  - Radionuclides decaying by pure beta transition ?

Method 4 π-γ

- Well suited for complex decay patterns: efficiency ≈ 100%
- Require proper electronic for signal processing and pile up
- Requiere a monte carlo model for precise corrections

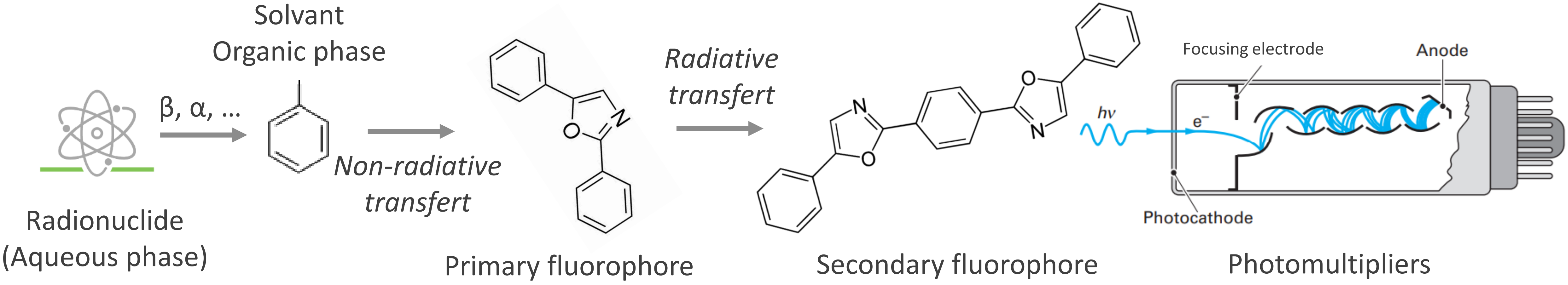


Non-exhaustive list of radionuclides measured at the LNHB by the 4π-γ : ^{18}F, ^{22}Na, ^{24}Na, ^{46}Sc, ^{56}Mn, ^{56}Co, ^{60}Co, ^{67}Cu, ^{67}Ga, ^{75}Se, ^{87m}Sr, ^{88}Y, ^{110m}Ag, ^{111}In, ^{124}Sb, ^{133}Ba, ^{134}Cs, ^{139}Ce, ^{152}Eu, ^{154}Eu, ^{166m}Ho, ^{169}Yb, ^{176}Lu, ^{192}Ir ^{203}Hg, ^{207}Bi, etc…

Liquid scintillation measurement, Triple to Double Coincidence Ratio (TDCR) method

Liquid scintillation

- **Use an aliquot of radioactive solution in a liquid scintillator (LS) prepared by weight measurement**
- **The liquid scintillator has the property to emit light in the presence of radiation**



- **Count the number of light pulses per unit of time using photomultipliers and adapted electronics**
- **Calculating detection efficiency: beware of quenching**
- **Deduce the activity of the source (or the mass activity of the solution)**

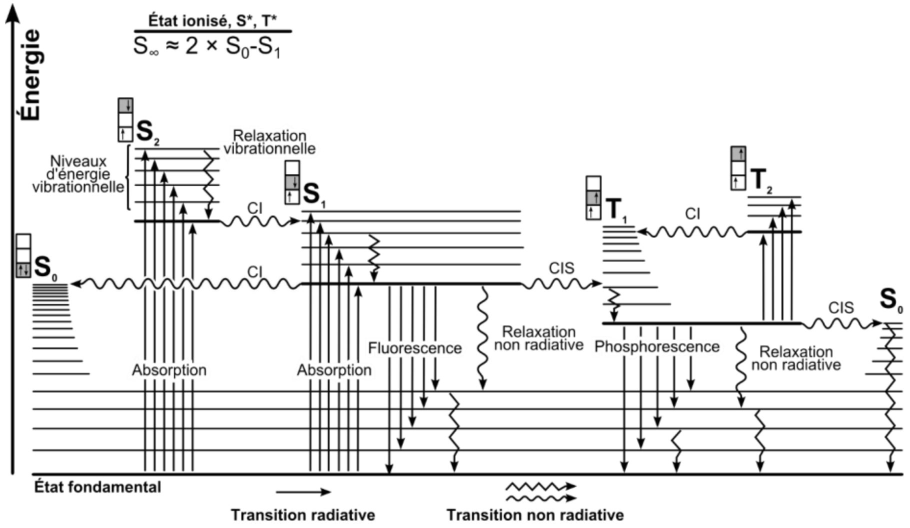
LSC as a direct measurement method

- **Detection efficiency *ε* is calculated without using a standard and the activity *A* is calculated with the count rate N (s^{-1})**

$$A = \frac{N}{\varepsilon}$$

- **ε is calculated from a model of the various physic-chemical phenomena occurring during the LSC process:**
  - radionuclide decay, radiation emission, radiation absorption by the scintillator, energy transfer in the scintillator, light emission statistics, light propagation, light detection by the photomultiplier tubes, coincidence counting…

- **There are a lot of transfers involved in the process:**
  - Light emission is a fast process (some ns) triplet states (µs)
  - Light emission yield is low (one keV of energy produces a few photons)
  - The number of photons emitted is random
  - The mean number of photons emitted is not proportional to the energy



Method widely used in National Metrology Institutes

- Measurand: activity of radioactive solution of a scintillating source prepared by weighing a few Bq to a few kBq
- Target radionuclides: pure beta emitters or electron capture to a fundamental level of the daughter nucleus, alpha emitters:
  - ^{3}H, ^{14}C, ^{18}F, ^{32}P, ^{33}P, ^{35}S, ^{36}Cl, ^{45}Ca, ^{51}Cr, ^{54}Mn, ^{55}Fe, ^{59}Ni, ^{60}Co, ^{63}Ni, ^{68}Ga, ^{68}Ge, ^{85}Kr, ^{89}Sr, ^{90}Sr, ^{90}Y, ^{99}Tc, ^{103}Pd, ^{109}Cd, ^{113}Sn, ^{125}I, ^{129}I, ^{137}Cs, ^{204}Tl, ^{210}Pb, ^{210}Po, ^{222}Rn, ^{226}Ra, ^{147}Pm, ^{153}Sm, ^{169}Er, ^{238}Pu, ^{239}Pu, ^{240}Pu, ^{241}Pu, ^{242}Pu, ^{241}Am
- Easy measurement if the detection efficiency is high and the decay pattern is simple
- Detection efficiency between < 0.5 and 1
- Primary measurement method for liquid scintillation: Triple to Double Coincidence Ratio TDCR, relative standard uncertainty of the measurement result: 0.2% to 1%.

Primary measurement technique for liquid scintillation: TDCR

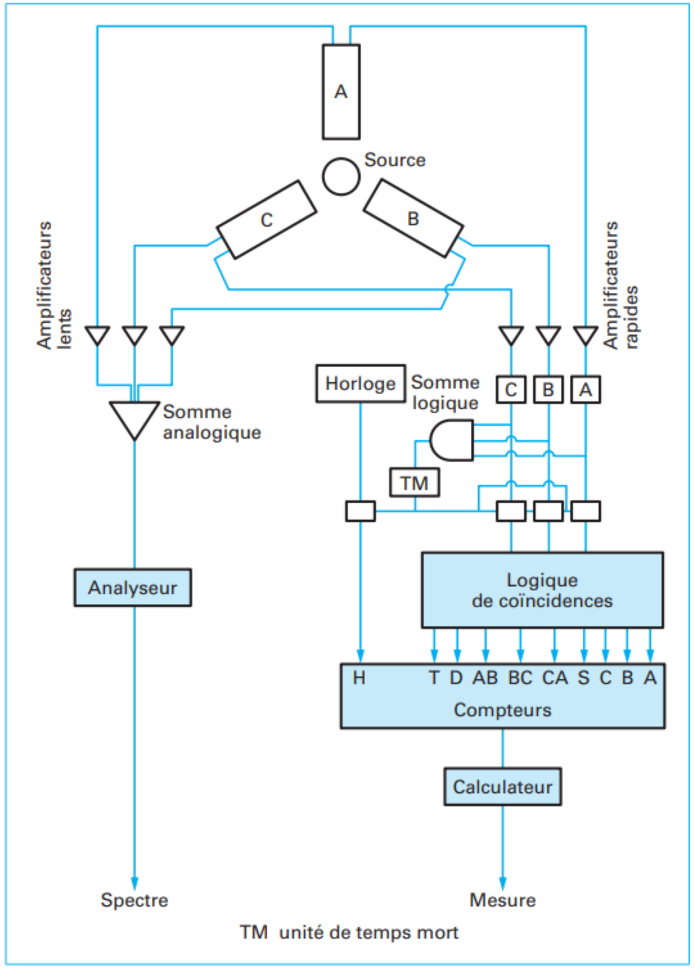
RCTD1 LNHB

- **National metrology institutes develop methods for the primary measurement of activity by liquid scintillation, enabling the production of national standards**
- **The TDCR method is based on a device using 3 PMTs for liquid scintillation measurements:**
  - One commercial device also available
  - Preferably: use your own device with known electronics in order to perform the measurement
- **Widely used in many field: medical, energy, …**
  - Target: α, β, *ec* nuclides

µ-TDCR
Portable device LNE-LNHB

B. Sabot, C. Dutsov, P. Cassette, K. Mitev, *Performance of portable TDCR systems developed at LNE-LNHB*, Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment, Volume 1034, 2022, 166721, ISSN 0168-9002, https://doi.org/10.1016/j.nima.2022.166721.

Implementation of the method



Schematic diagram of the meter
(P. Cassette, Technique de l'ingénieur, P2552 v2)

- **Requires a scintillation counter with 3 photomultipliers (PMTs)**
  - 1/3 symmetry: 120° between each PMT
- **Measurement geometry: typical 20 mL liquid scintillation vial filled to 10 mL with scintillating liquid**
- **Electronics to acquire the average number of triple (T) and double (D) coincidences but also:**
  - each individual signal (A, B and C) with a threshold setting
  - each double coincidence (AB, BC, BC)
- **Detection efficiency calculated from the T/D ratio**
- **CAUTION: The calculation is based on three fundamental assumptions**

Description

- **Assumption 1: for a deposit of energy *E* in the scintillator, the probability of emission of *x* photons for an average number *m* follows a Poisson distribution:**

$$P\,(x/m) = \frac{m^x\, e^{-m}}{x!}$$

- **Assumption 2: the probability of detection of a photon is not zero, the detection efficiency is:**

$$1 - P\,(0/m) = 1 - e^{-m}$$

For a 3 PMs detector in 1/3 symmetry of quantum efficiency *v*:

Efficiency for 1 photodetector	$R_1 = 1 - e^{-\frac{vm}{3}}$
Efficiency for 2 photodetectors in coincidence	$R_2 = \left(1 - e^{-\frac{vm}{3}}\right)^2$
Efficiency for 3 photodetectors in coincidence	$R_T = \left(1 - e^{-\frac{vm}{3}}\right)^3$
Return for the logical sum of double coincidences	$R_D = 3\left(1 - e^{-\frac{vm}{3}}\right)^2 - 2\left(1 - e^{-\frac{vm}{3}}\right)^3$

- **Assumption 3: the light yield follows Birks law giving the average number of photons emitted for the absorption in the scintillator of an energy *E*:**

$$m(E) = \int_0^E \frac{\alpha\, dE}{1 + k_B \frac{dE}{dx}}$$

With α scintillation efficiency (photons·keV^{-1}), k_B scintillator parameter (from 0.005 to 0.018 cm·MeV^{-1}), *dE/dx* the linear energy transfer of ionizing radiation

- **Considering the normalized spectrum *S(E)* of the energy emitted by the radionuclide**

$$TDCR = \frac{\int_{spectrum} S(E)\left(1 - e^{-\frac{\nu\alpha m(E)}{3}}\right)^3}{\int_{spectrum} S(E)\left(3\left(1 - e^{-\frac{\nu\alpha m(E)}{3}}\right)^2 - 2\left(1 - e^{-\frac{\nu\alpha m(E)}{3}}\right)^3\right)} \quad where\ m\,(E) = \int_0^E \frac{dE}{1 + k_B \frac{dE}{dx}}$$

- **Considering that for a large number of events, the ratio of frequencies converges to the ratio of probabilities, the computational algorithm consists in finding the free parameter, $\nu\alpha$, such that:**

$$TDCR = \frac{T}{D} = \frac{\varepsilon_T}{\varepsilon_D}$$

In practice

- **PMTs are never exactly identical:**

$$\frac{\varepsilon_T}{\varepsilon_{AB}} = \frac{\int_{spectrum} S(E)\left(1-e^{-\frac{\nu_A \alpha m(E)}{3}}\right)\left(1-e^{-\frac{\nu_B \alpha m(E)}{3}}\right)\left(1-e^{-\frac{\nu_C \alpha m(E)}{3}}\right)\mathrm{dE}}{\int_{spectrum} S(E)\left(1-e^{-\frac{\nu_A \alpha m(E)}{3}}\right)\left(1-e^{-\frac{\nu_B \alpha m(E)}{3}}\right)dE}$$

Similar for $\frac{\varepsilon_T}{\varepsilon_{BC}}$ and $\frac{\varepsilon_T}{\varepsilon_{AC}}$

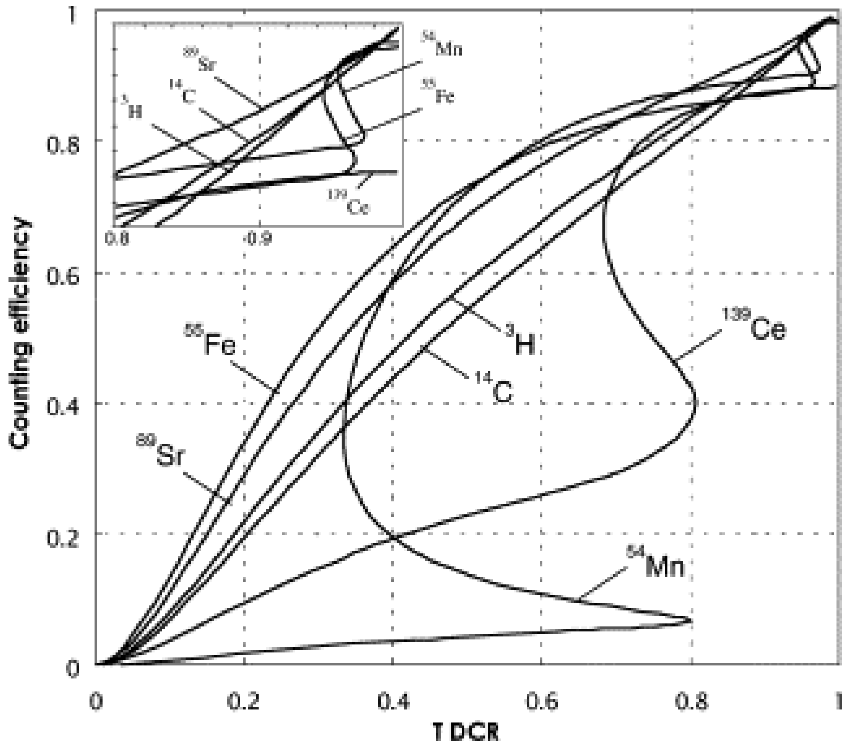
- **The solution to be minimized becomes:**

$$\Delta = \left(\frac{\varepsilon_T}{\varepsilon_{AB}} - \frac{R_T}{R_{AB}}\right)^2 + \left(\frac{\varepsilon_T}{\varepsilon_{BC}} - \frac{R_T}{R_{BC}}\right)^2 + \left(\frac{\varepsilon_T}{\varepsilon_{AC}} - \frac{R_T}{R_{AC}}\right)^2$$

K. Kossert, B. Sabot, P. Cassette, R. Coulon, H. Liu, On the photomultiplier-tube asymmetry in TDCR systems, 2020, Appl Radiat Isot 163, 109223, https://10.1016/j.apradiso.2020.109223/

- **A single solution for electrons**

- **Up to 3 solutions for β-γ emitters and electron capture: the solution to remove ambiguity is to vary the yield**



Evolution of yield as a function of TDCR for different radionuclides

R. Broda, A review of the triple-to-double coincidence ratio (TDCR) method for standardizing radionuclides, 2003, Applied Radiation and Isotopes, Volume 58, Issue 5, May 2003, Pages 585-594 https://doi.org/10.1016/S0969-8043(03)00056-3

TDCR measurement example

High dependency on the nuclear data ! Example with pure beta emitter ^{63}Ni

- **Comparison between 2 LSC methods demonstrates the importance of precise β spectrum**
  - Screening and exchange added in the spectrum calculation

➔ Limit of this case: only LS measurement

Applied Radiation and Isotopes 101 (2015) 40–43

Contents lists available at ScienceDirect

Applied Radiation and Isotopes

journal homepage: www.elsevier.com/locate/apradiso

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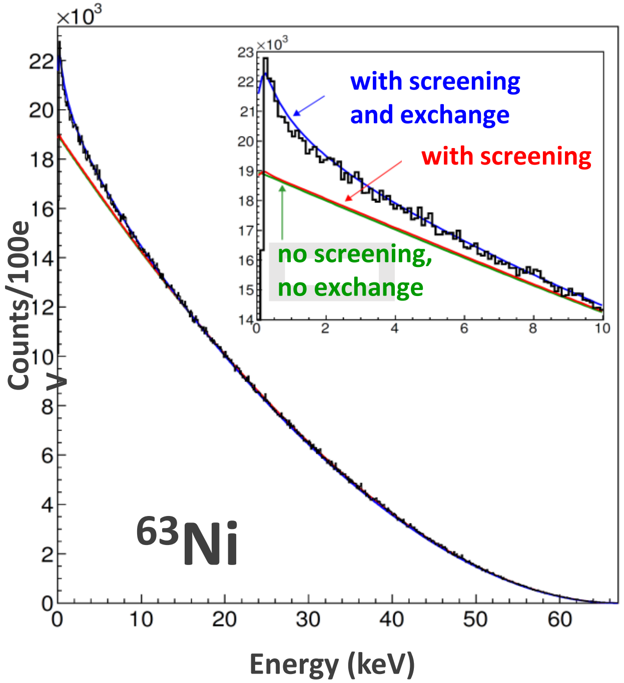
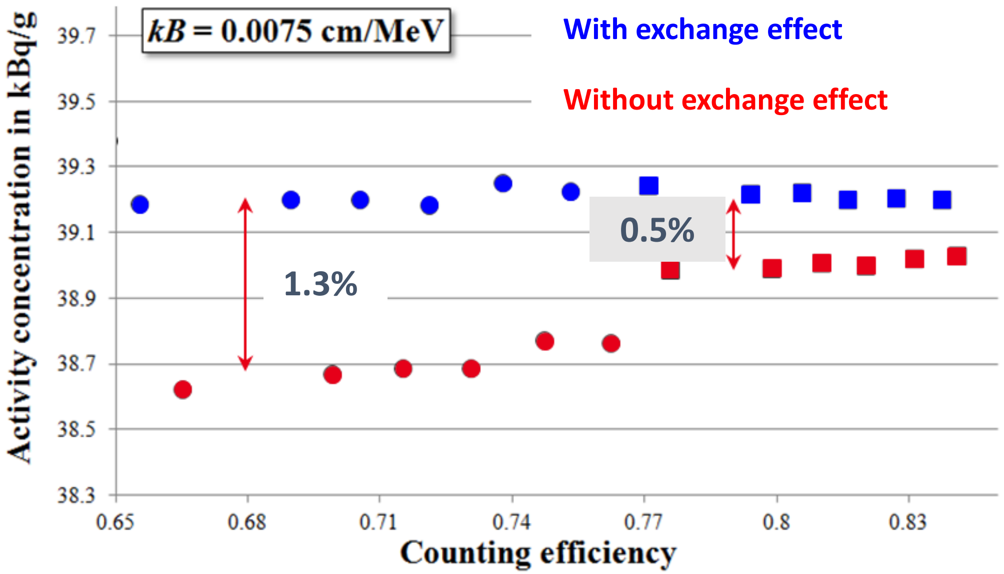
The importance of the beta spectrum calculation for accurate activity determination of ^{63}Ni by means of liquid scintillation counting

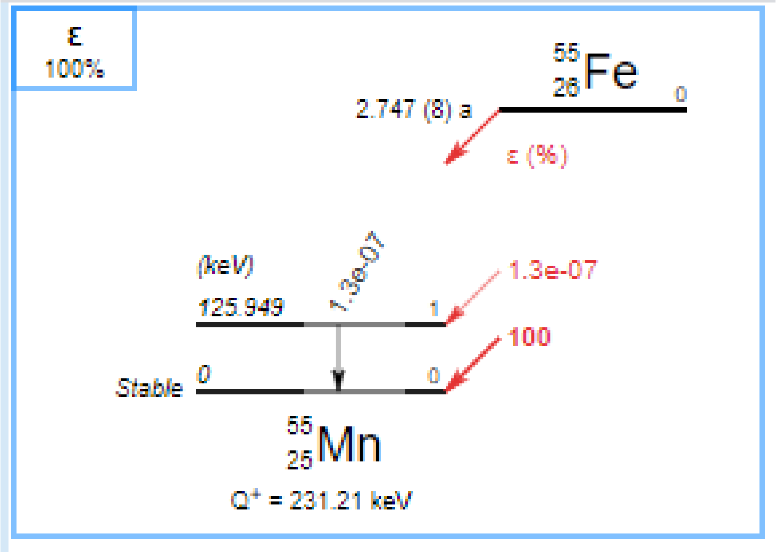
Karsten Kossert [a,*], Xavier Mougeot [b]





Simple electron capture with low energy: ^{55}Fe

- **Measurement performed in the framework of CCRI(II)-K2.Fe-55.2019**
  - Activity concentration measurements of a ^{55}Fe solution
  - Organized by Polatom with 11 NMIs
  - All measurements were performed by liquid scintillation either TDCR or CIEMAT NIST method
- **Side note: diffusive vials were used**



CCRI(II)-K2.Fe-55.2019

Final Report of the key comparison CCRI(II)-K2.Fe-55.2019 of activity concentration measurements of a ^{55}Fe solution

R. Broda[1], I. Bonková[7], M. Capogni[2], P. Carconi[2], P. Cassette[4], R. Coulon[3], S. Courte[3], P. De Felice[2], T. Dziel[1], A. Fazio[2], C. Frechou[4], R. Galea[5], E. García-Toraño[10], E. Kołakowska[1], K. Kossert[6], M. Krivošík[7], E. Lech[1], K.B. Lee[8], J. Liang[9], A. Listkowska[1], H. Liu[9], N. Navarro[10], O.J. Nähle[6], M. Nowicka[1], M. van Rooy[11], B. Sabot[4], P. Saganowski[1], Y. Sato[12], Z. Tymiński[1], A. Yunoki[12], M. Zhang[9], T. Ziemek[1].

Measurement of Fe-55 with SL-TDCR

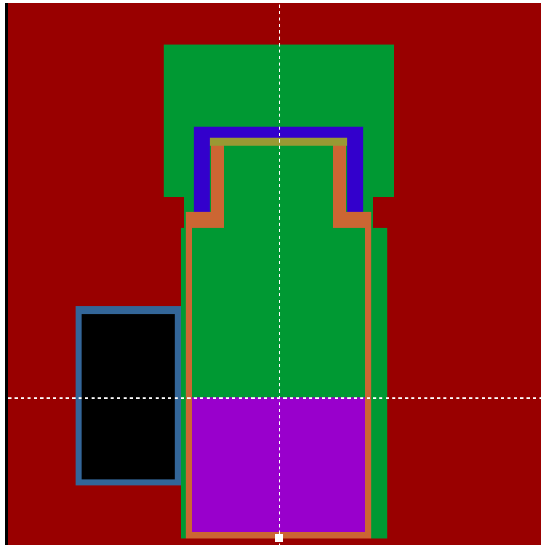
				Absorbed energy	Probability
K	XK_{α}	Absorbed	X_L/Auger L	K_{α} (Compton+photoeletric)+X_L/A_L	$P_K*\omega_K*RK_{\alpha}*PK_{\alpha}/PK_{tot}$
		Escaped	X_L/Auger L	X_L/A_L	$P_K*\omega_K*(1-RK_{\alpha})*PK_{\alpha}/PK_{tot}$
	XK_{β}	Absorbed		K_{β} (Compton+photoeletric)	$P_K*\omega_K*RK_{\beta}*PK_{\beta}/PK_{tot}$
		Escaped		0	$P_K*\omega_K*(1-RK_{\alpha})*PK_{\alpha}/PK_{tot}$
	Auger KLL	X_L/Auger L	XL/Auger L	$A_{KLL}+X_L/A_L+X_L/A_L$	$P_K*(1-\omega_K)*P_{KLL}/P_{Ktot}$
	Auger KLX	X_L/Auger L		$A_{KLX}+X_L/A_L$	$P_K*(1-\omega_K)*P_{KLX}/P_{Ktot}$
	Auger KXY			A_{KXY}	$P_K*(1-\omega_K)*P_{KXY}/P_{Ktot}$
L	XL/Auger L			X_L/A_L	P_L
M, N				0	$1-(P_K+P_L)$

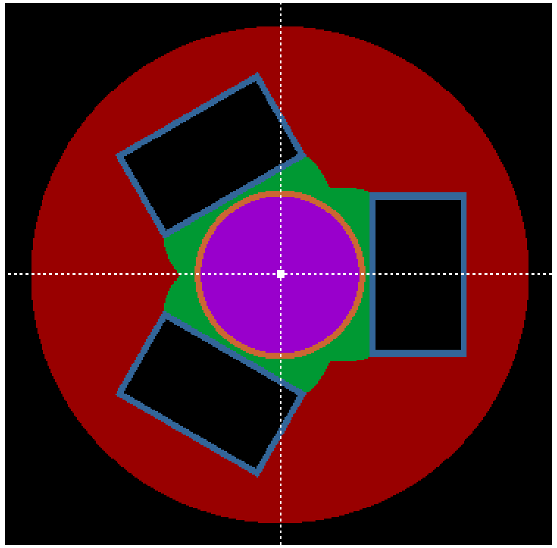
^{55}Fe decay path

- For electron capture the knowledge of decay path is very important
- A model can be constructed with some assumptions depending on the energies and probabilities
- In the case of ^{55}Fe the effect of M, N and even L events have a very small influence but this is not the case for all the nuclides
- For such measurement a Monte-Carlo model is required in order to get the probability absorption of X rays

Monte-Carlo model

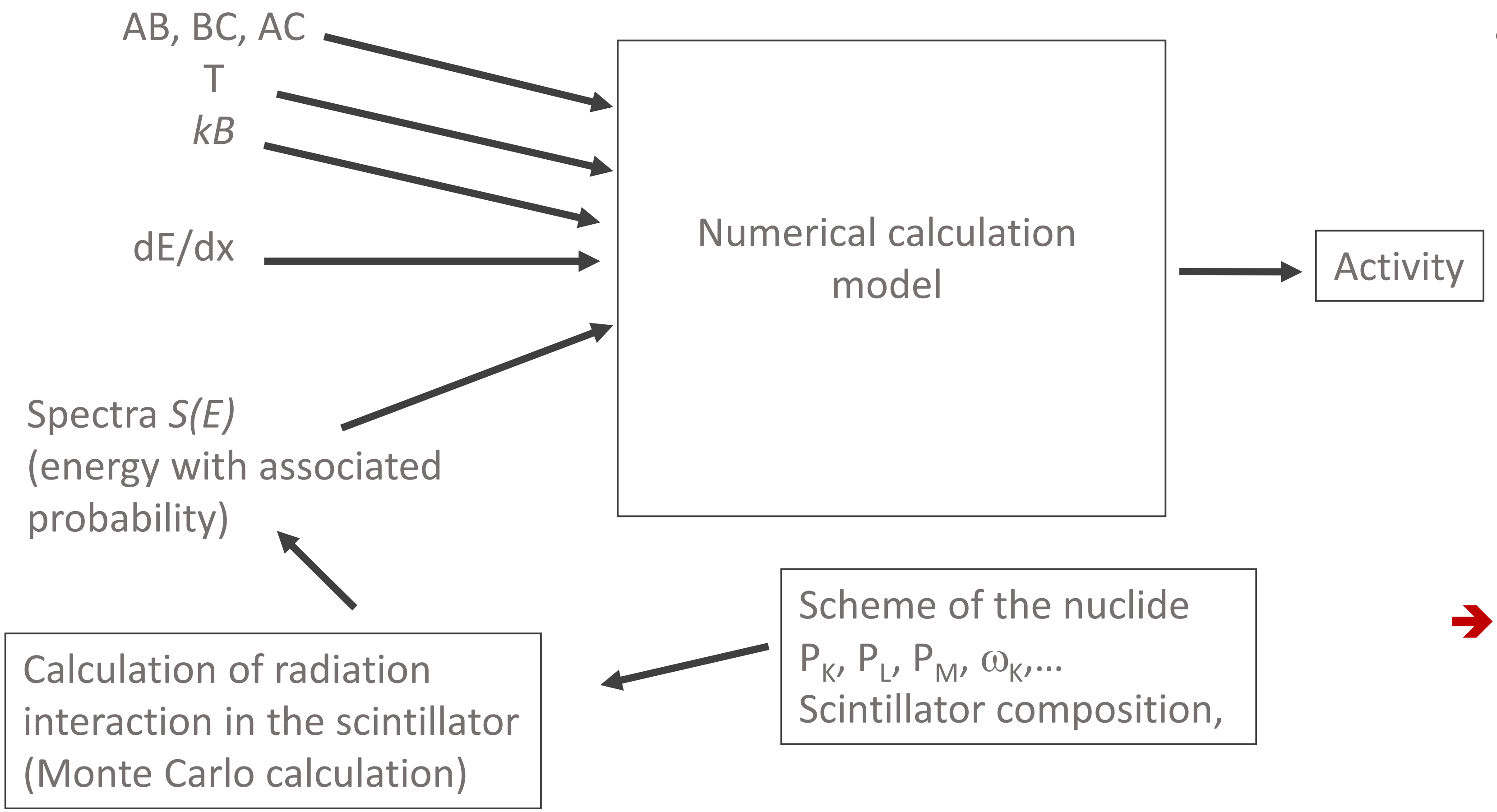
- **The TDCR counter is simulated with the Monte-Carlo code Penelope 2018 (Salvat *et al.*)**
  - Composition of the liquid scintillator is an important input parameter
- **It can be a very precise model which takes also into account other parameters of the device (PMTs, walls of the chamber…)**
  - Not important in the case of ^{55}Fe but critical for higher energy photons





→Nuclear data is an input of Penelope code and it is also very important to use proper data

Activity computation with SL-TDCR measurements

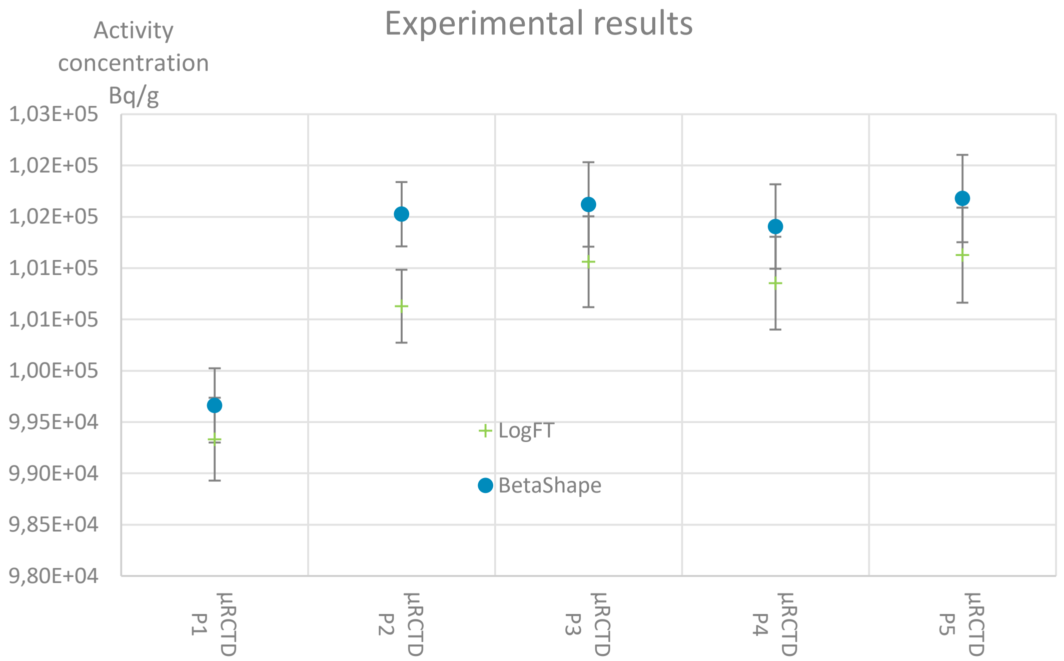


- **Uncertainty budget on the activity calculation is propagated by Monte Carlo method:**
  - The calculations are repeated with n number of events
  - The result is the average of the n calculations
  - We can get the standard deviation and the distribution of the results

➔ **Strong dependence on the nuclide data**

Mass activity measurements with µ-TDCR

- **Around 100 mg of the ^{55}Fe solution were placed into a diffusive LS vial with 10 mL of UGAB:**
  - 5 samples were prepared
  - The activity was calculated using old data and also improved calculation of fractional electron-capture probabilities by X. Mougeot



- **0.6% of relative deviation between both set of results**

➔ Clear experimental impact of the data but there is no comparison with another measurement method for ^{55}Fe

Summary

Summary

The purpose of metrology is to establish reference methods and standards and have homogeneous units throughout the world

- **In radionuclide metrology there is not an absolute method to define the Bq unit**
- **Polymorphism of radionuclide makes it very difficult to measure**
- **Many methods were developed sometimes you can measurement more than one nuclide sometime only one with a method**
  - Coincidence
  - 4π well detecto
  - Define solide angle method
  - TDCR with liquid scintillation
  - And more…. Compton-TDCR soon ? ☺
- **The important point is to produce standards to transfer them to users and to allow an harmony of units and measurements throughout the world thanks to the research work of national and international metrology institutes**

Thanks

This project has received funding from the European Union’s Horizon 2020 research and innovation programme under grant agreement No 899293