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International Centre for Theoretical Physics



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**Workshop on Understanding and Evaluating Radioanalytical
Measurement Uncertainty**

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**Combined Uncertainty in the determination of ^{90}Sr by Liquid Scintillation
Counting - LSC**

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Combined Uncertainty in the determination of ^{90}Sr by LSC

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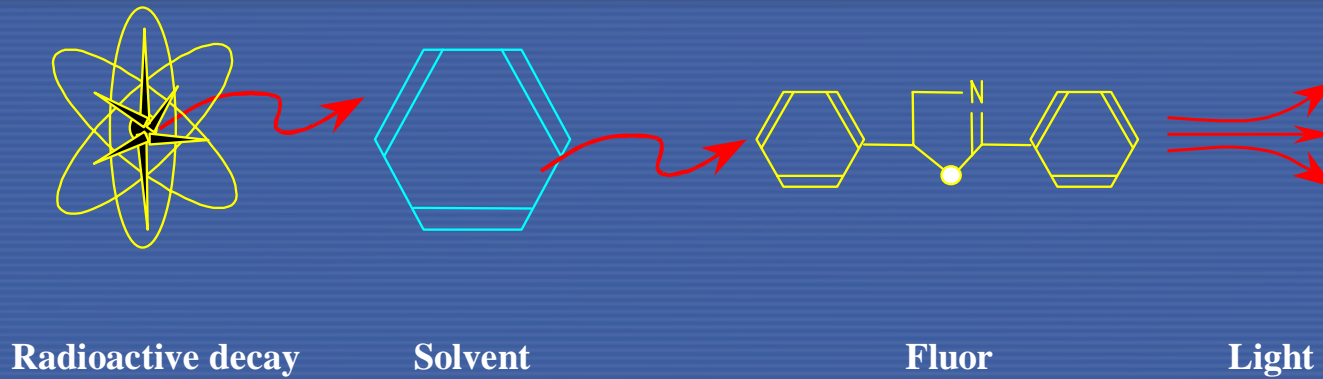
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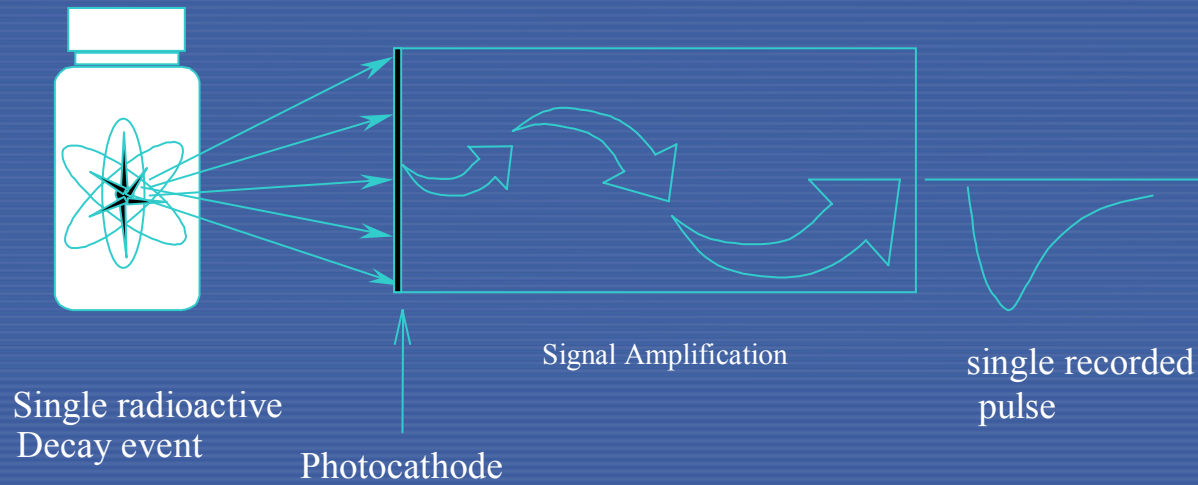
Contents

- **Introduction of LSC**
 - ✓ Liquid scintillation process
 - ✓ Quenching
 - ✓ Background
- **Calculation of ^{90}Sr activity concentration and its combined uncertainty**
 - ✓ Tailing factor
 - ✓ Chemical recovery and counting efficiency
 - ✓ Statistics of count rate
 - ✓ Ash/wet weight ratio
- **Preparation of spread sheet**

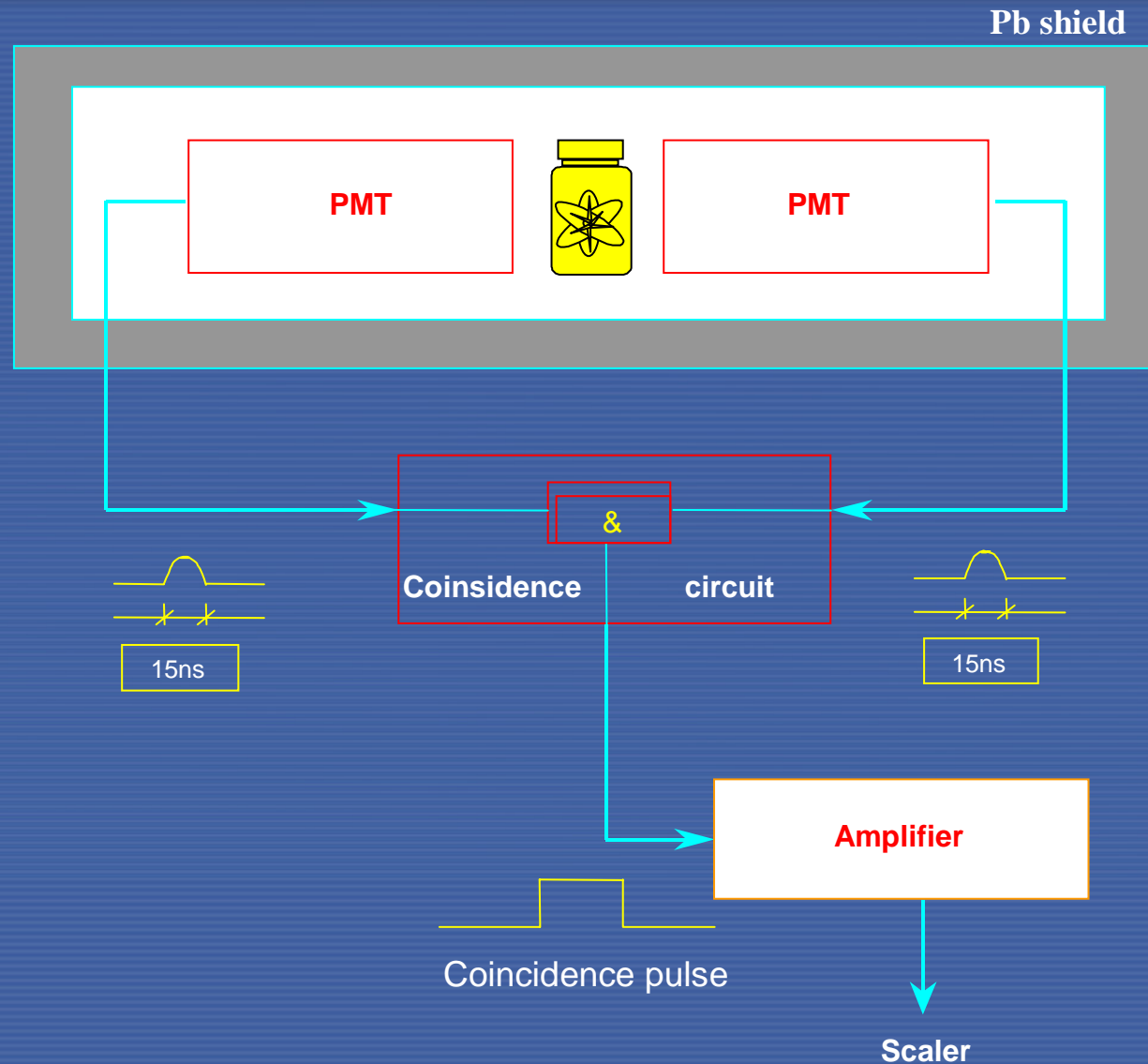
Liquid Scintillation Process



The energy transfer process



Block diagram of Liquid Scintillation Counter



Background in Liquid Scintillation Counter

- **Random coincidences** ; thermionic noise of PMT
- **Crosstalk** ; photon produced on the surface of glass of PMT by Cerenkov and electron discharge
- **Intrinsic Radioactivity of the Counter itself** (metallic parts, PMTs) and the vial and cocktail
- **Cosmic Radiation**
- **Environmental Gamma radiation** from ground bedrock and building materials, i.e. from ^{40}K , ^{238}U and ^{232}Th
- **Static electricity**
- **Chemiluminescence** ; photon produced by chemical energy

Liquid Scintillation Counter

□ Advantages

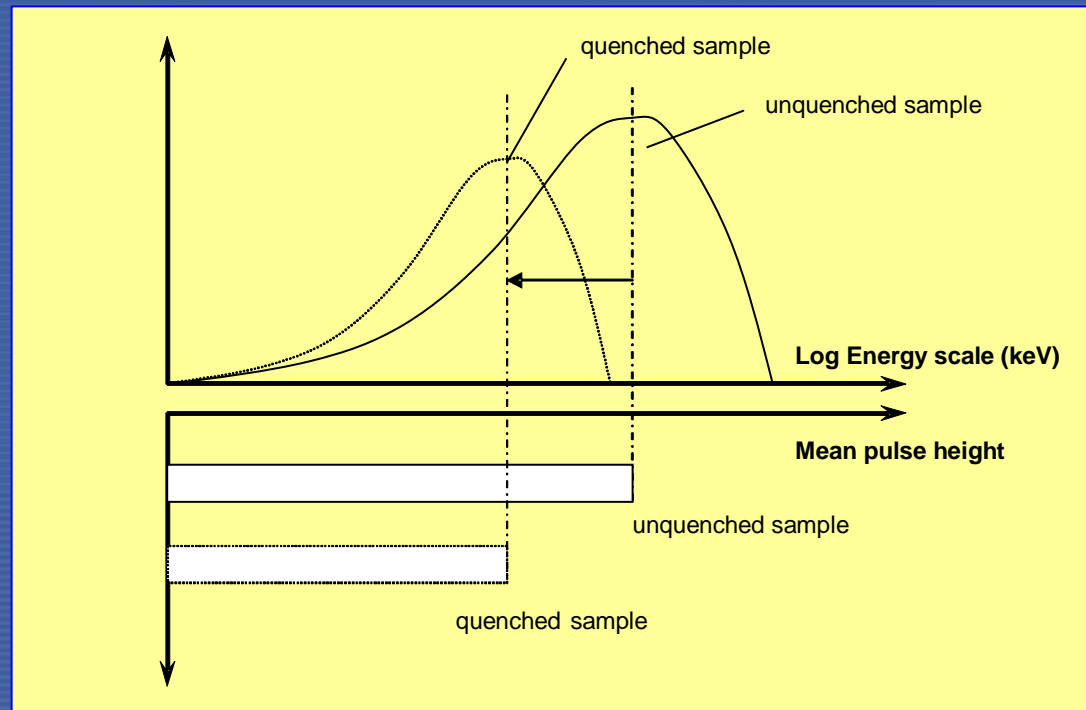
- ✚ Self-absorption is usually negligible
- ✚ No absorption of radiations by air or a detector's window
- ✚ No radiation scattering prior to incidence upon the detector
- ✚ High counting efficiency
- ✚ Easy sample preparation

□ Some shortcomings

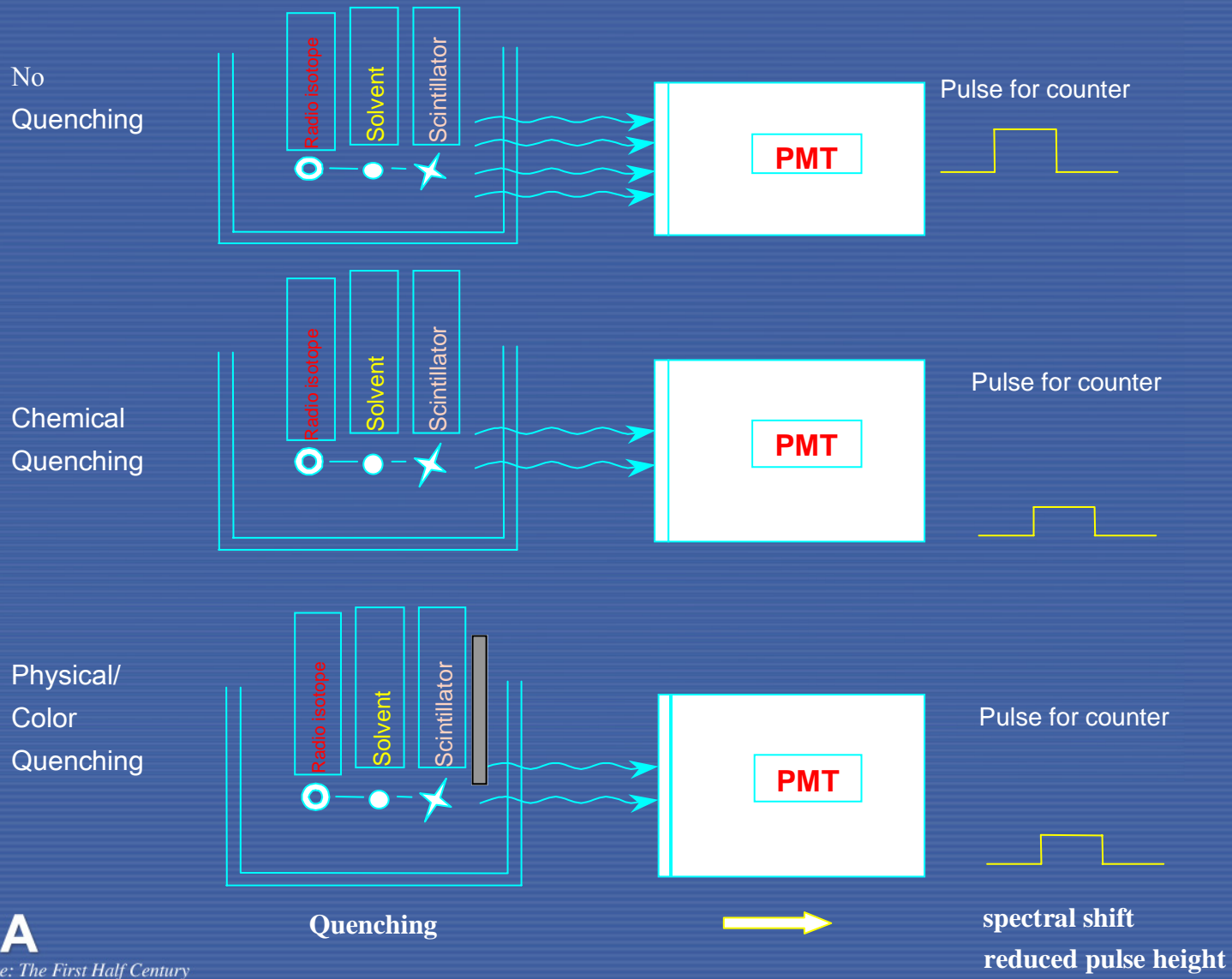
- ✚ Quenching effect
- ✚ Interference of chemiluminescence
- ✚ Production of organic radioactive waste
- ✚ Higher background than α -spectrometer

Quenching

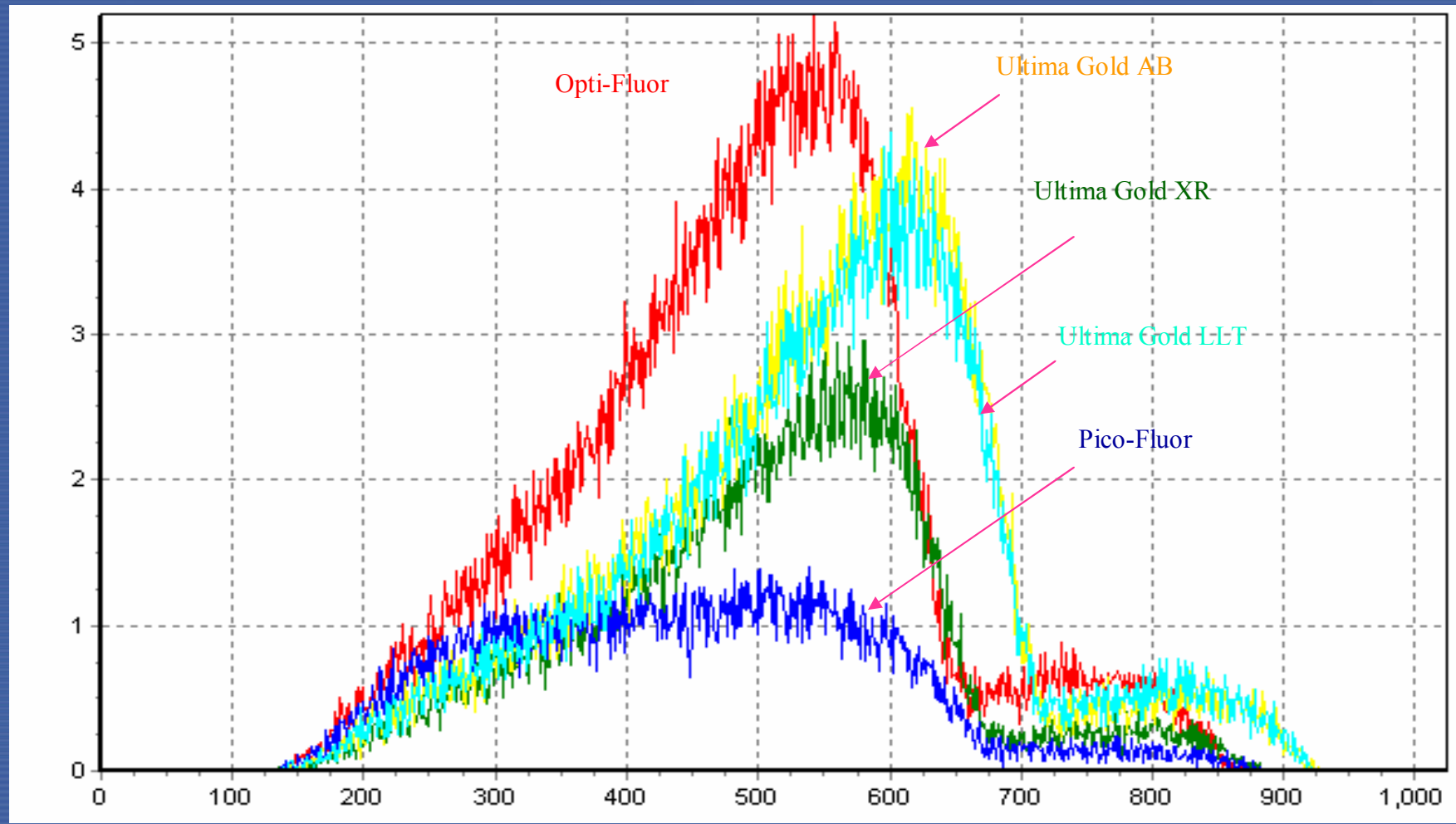
- **Quenching** causes
 - Reduction in the number of photons reaching photo-cathode
 - Reduction of the height of electrical pulse and number of counts or pulses
 - Shift of the spectrum toward low energy



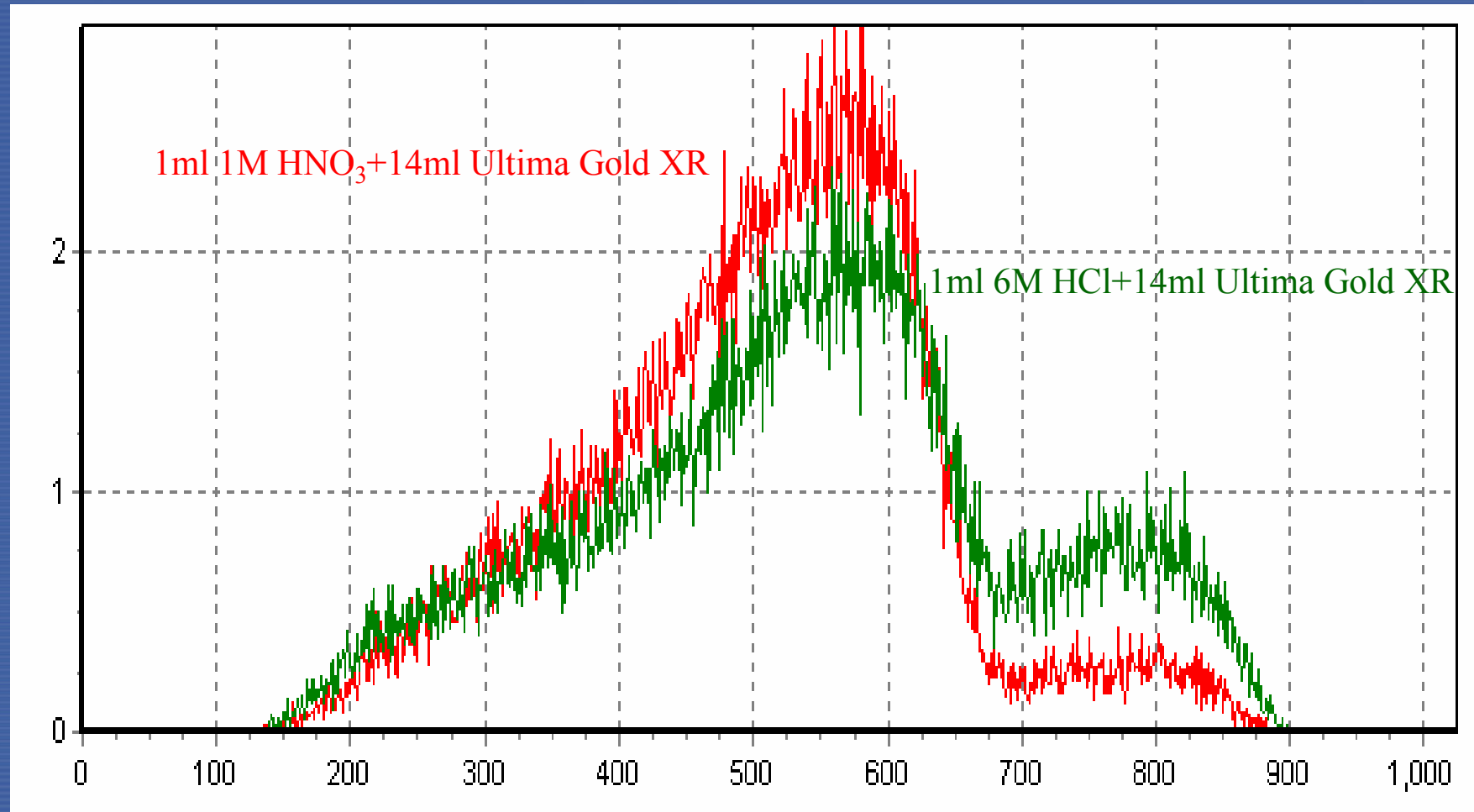
Different forms of quenching and effect of quenching on pulse height



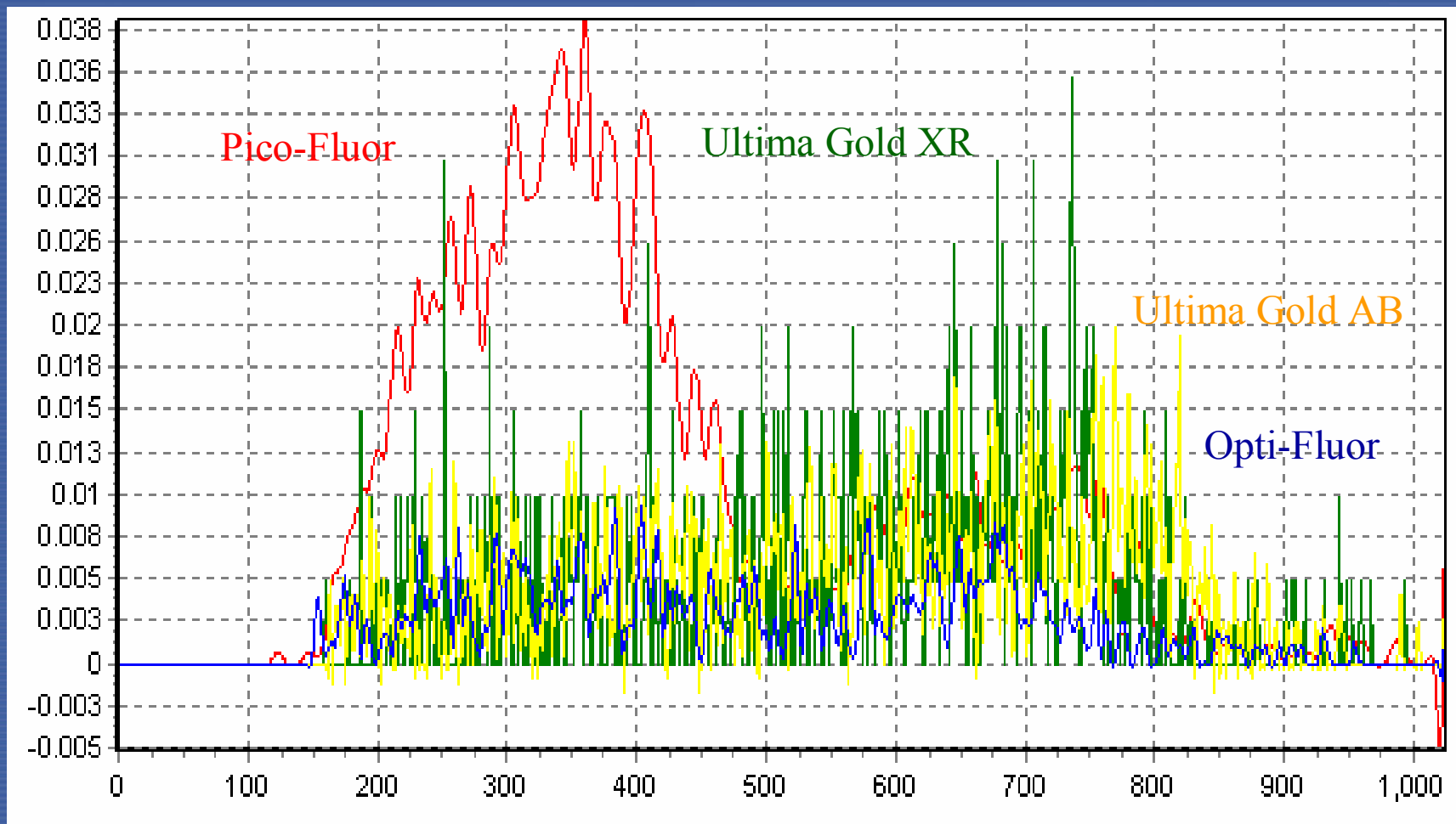
Quenching effect depending on different types of cocktails



Quenching effect depending on different types of solvents



Background variation depending on different types of solvents



How to calculate the combined uncertainty

Approach and structure



Specification

- Analytical task, equation to be used for the calculation of the result

Identify uncertainty sources

- List sources of uncertainty for each part of the process

Quantifying uncertainty components

- Estimate the size of each uncertainty component

Convert to standard uncertainty

- Obtain the standard uncertainty

Calculate the combined uncertainty

- Combine the uncertainty components

Report results and uncertainty

- Measurement result, its uncertainty



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Specifications

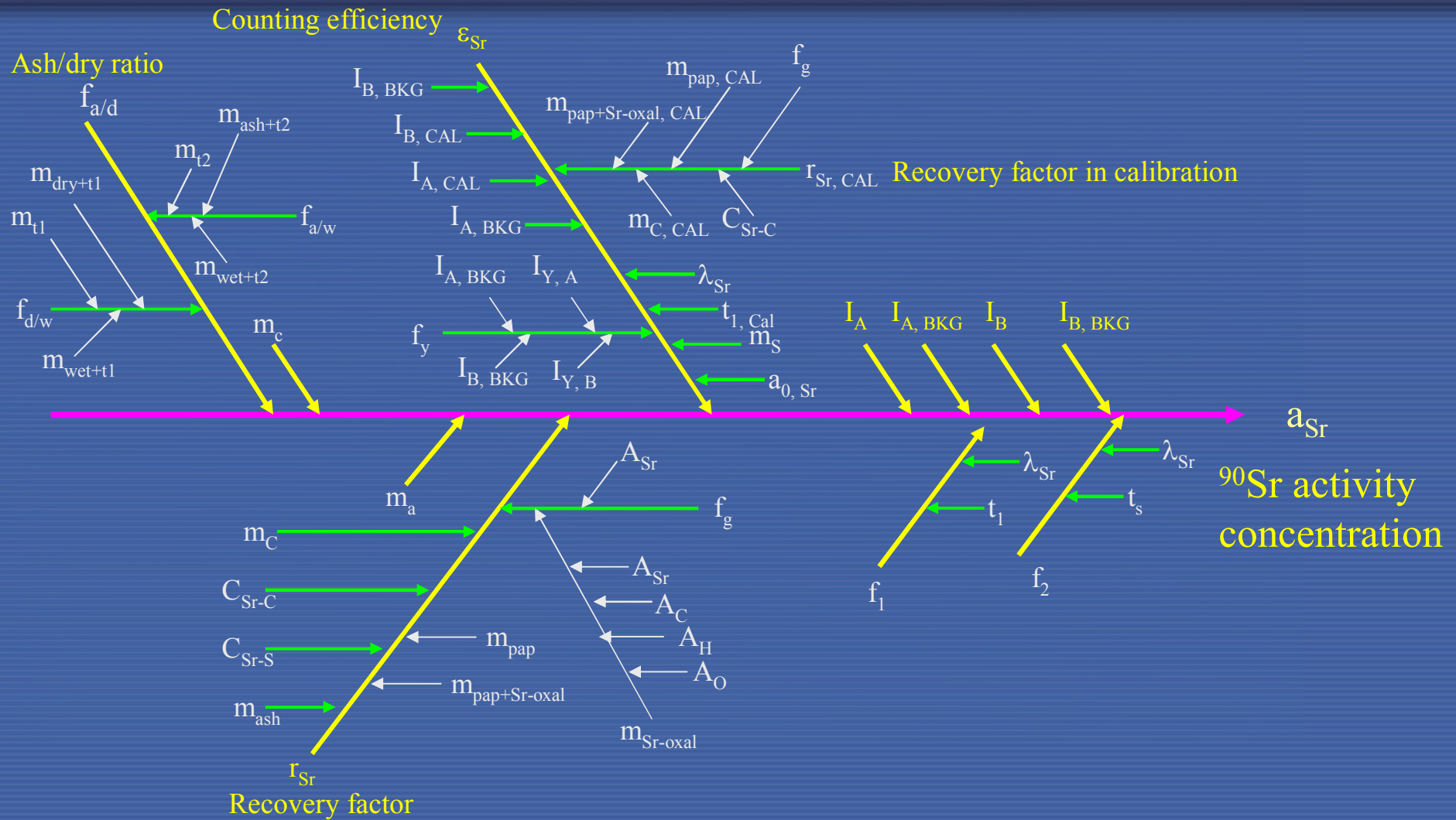
- Technique : LSC
- Sample : Soil
- Analyte : Sr-90
- Measurand : activity of Sr-90 in soil sample

Identify uncertainty sources

- Counting efficiency and Tailing factor (double window)
- Chemical recovery
- Weighing sample, Sr carrier and ^{90}Sr standard solution
- Count rates of the sample
- Counts rates of BKGs
- Decay correction of ^{90}Sr

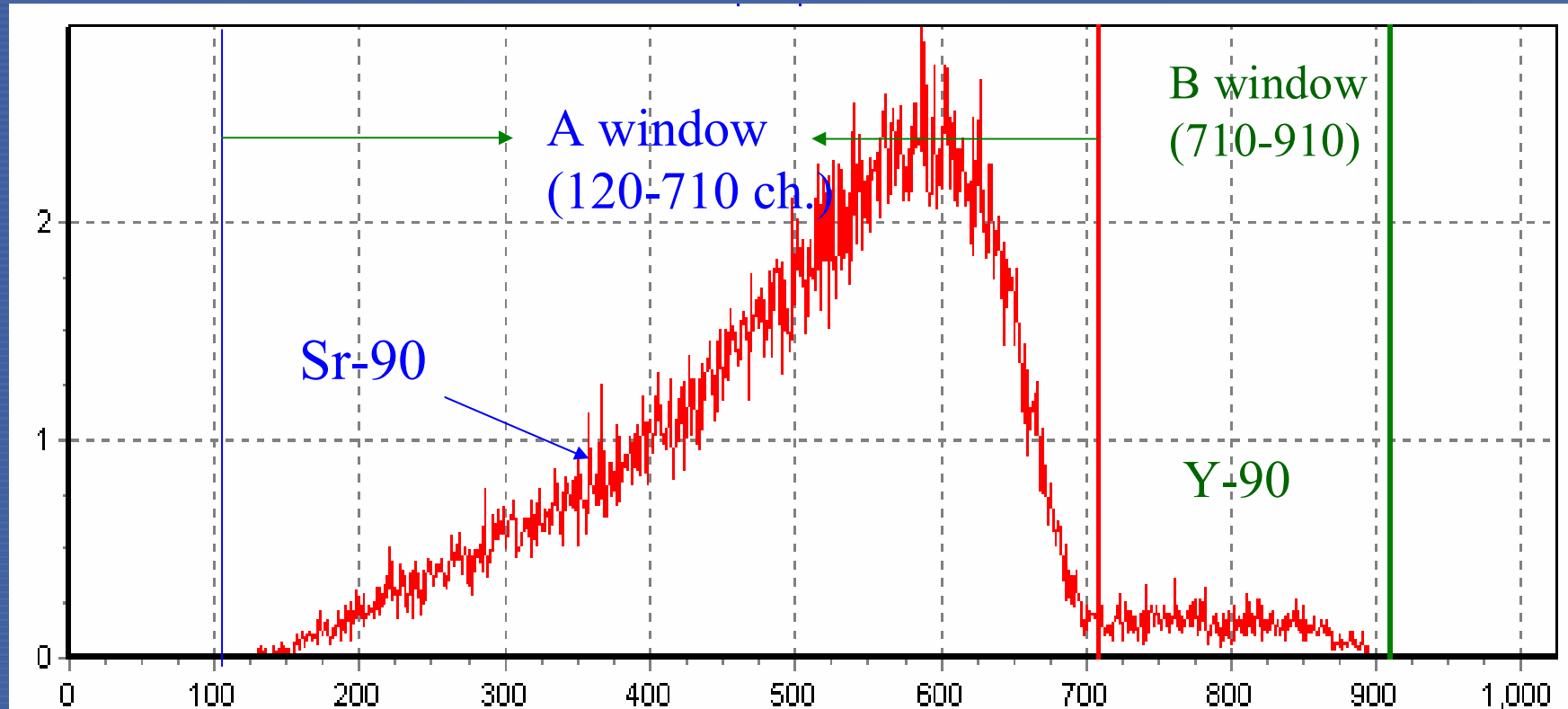


2.1. Cause and effect diagram for combined uncertainty in the determination of ^{90}Sr by LSC



1. Determination of Tailing factor (f_y)

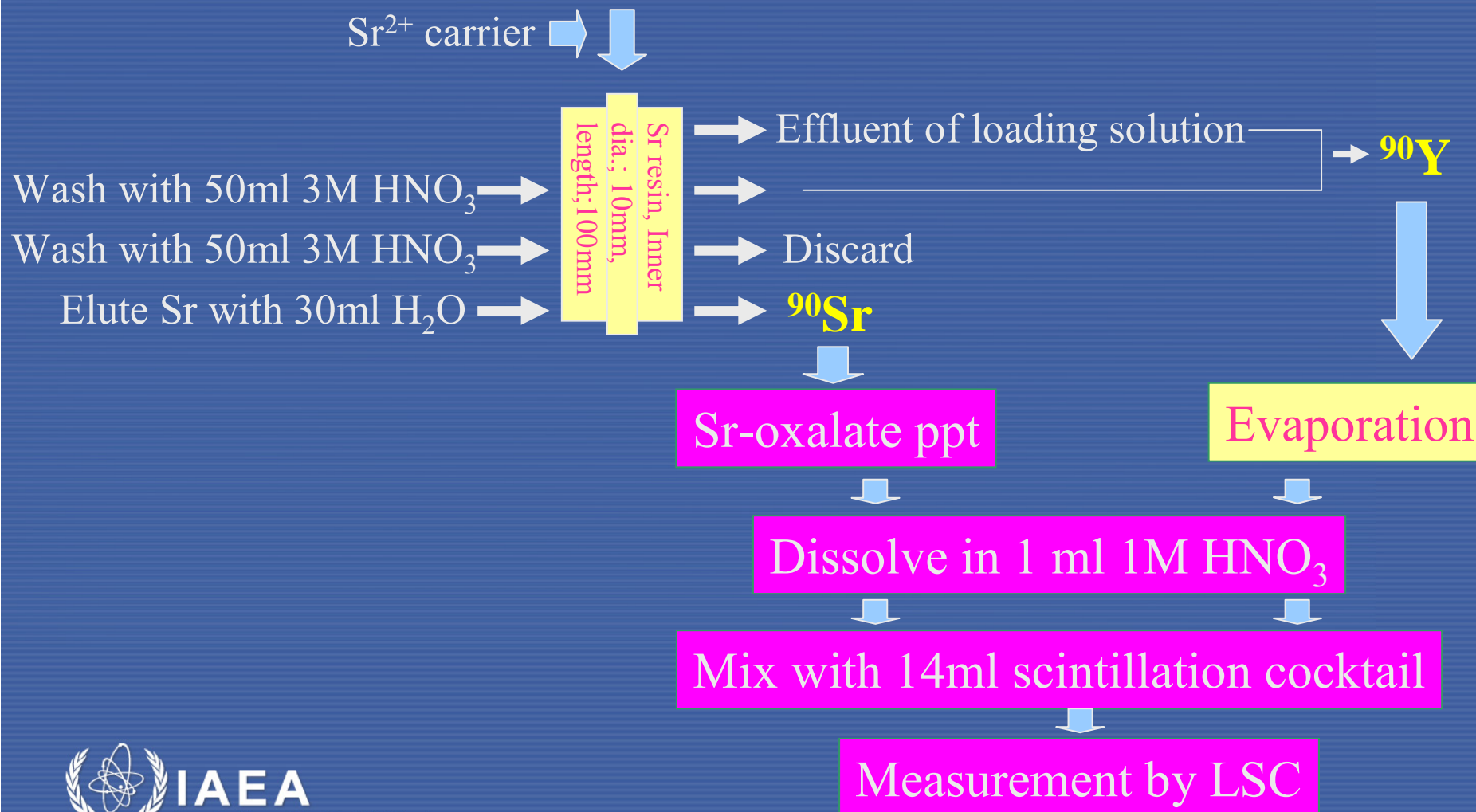
Spectra of Sr-90 calibration source



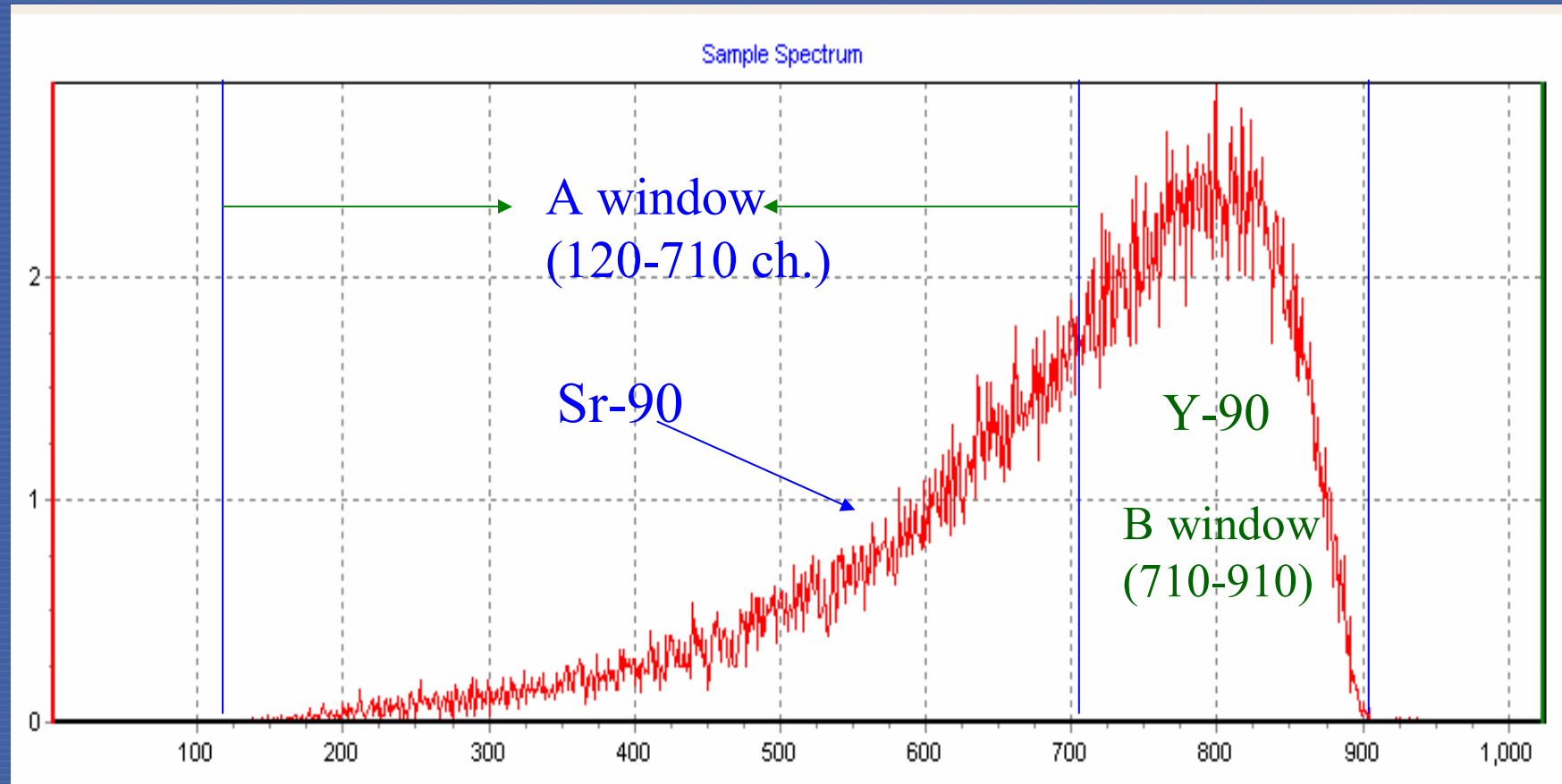
Net counts corrected in-growth Y-90 for Sr-90 = Net CPM in A win - f_y * Net CPM in B win

2.2. Radiochemical procedure of ^{90}Sr for calibration source preparation

Taking a known activity of ^{90}Sr standard solution



Y-90 spectrum



$$f_y = \text{Net CPM in A window} / \text{Net CPM in B window}$$

1.2. Tailing factor (f_y) and uncertainty

$$f_y = \frac{(I_{y,A,Cal} - I_{A,Bkg})}{(I_{y,B,Cal} - I_{B,Bkg})} \quad (1)$$

$$u_{f_y} = f_y \cdot \sqrt{\frac{(uI_{y,A,Cal}^2 + uI_{A,Bkg}^2)}{(I_{y,A,Cal} - I_{A,Bkg})^2} + \frac{(uI_{y,B,Cal}^2 + uI_{B,Bkg}^2)}{(I_{y,B,Cal} - I_{B,Bkg})^2}} \quad (2)$$

f_y : tailing factor

$I_{y,A,Cal}$: gross count rate of in window A of ^{90}Y calibration source spectrum [cpm]

$I_{y,B,Cal}$: gross count rate in window B of ^{90}Y calibration source spectrum [cpm]

$I_{A,Bkg}$: count rate of blank in window A [cpm]

$I_{B,Bkg}$: count rate of blank in window B [cpm]



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Practical exercise 1 – uncertainty of tailing factor

After separating ^{90}Y from 20Bq of ^{90}Sr standard by Sr resin extraction chromatography, ^{90}Y fraction was dissolved in 1ml 1M HNO_3 and mixed with 14ml Instagel cocktail. The reagent blank also prepared by the same way. The samples were 3 replicates measured by LSC for 30 min for each sample. Assuming that the following data are observed, **please calculate tailing factor and its uncertainty.**

| Sample name | replicate No. | Gross count rate (cpm) | | | |
|---------------|---------------|------------------------|-------------------|------------------|-------------------|
| | | window A (ly, A) | $\mu\text{ly, A}$ | window B (ly, B) | $\mu\text{ly, B}$ |
| STD-1 | 1 | 253.800 | 2.070 | 369.900 | 2.499 |
| | 2 | 235.500 | 1.994 | 349.300 | 2.429 |
| | 3 | 218.500 | 1.921 | 318.600 | 2.319 |
| Reagent blank | 1 | 4.053 | 0.262 | 1.672 | 0.168 |
| | 2 | 3.597 | 0.246 | 1.638 | 0.166 |
| | 3 | 3.664 | 0.249 | 1.655 | 0.167 |

Solution for Practical exercise 1

| Sample name | replicate No. | Gross count rate (cpm) | | | |
|---------------|---------------|------------------------|--------------|-----------------------|--------------|
| | | window A (l_y, A) | $\mu l_y, A$ | window B (l_y, B) | $\mu l_y, B$ |
| STD-1 | 1 | 253.800 | 2.070 | 369.900 | 2.499 |
| | 2 | 235.500 | 1.994 | 349.300 | 2.429 |
| | 3 | 218.500 | 1.921 | 318.600 | 2.319 |
| Reagent blank | 1 | 4.053 | 0.262 | 1.672 | 0.168 |
| | 2 | 3.597 | 0.246 | 1.638 | 0.166 |
| | 3 | 3.664 | 0.249 | 1.655 | 0.167 |

| Sample name | window A | | windowB | |
|---------------|--------------------------|---------------------------|--------------------------|--------------------------|
| | Average(l_y, A, Bkg) | STV ($\mu l_y, A, Bkg$) | Average(l_y, B, Bkg) | STV($\mu l_y, B, Bkg$) |
| Reagent blank | 3.771 | 0.246 | 1.655 | 0.017 |

| Sample name | Net count rate (cpm) | | Tailing factor | | Average, f_y | STV |
|-------------|----------------------|---------|----------------|-----------|----------------|--------------|
| | window A | windowB | f_y | μf_y | | |
| STD-1 | 250.029 | 368.245 | 0.67897 | 0.00730 | | |
| | 231.729 | 347.645 | 0.66657 | 0.00742 | | |
| | 214.729 | 316.945 | 0.67750 | 0.00787 | 0.674 | 0.008 |

2. Determination of chemical recovery in ^{90}Sr calibration source preparation

2.1. Chemical recovery of Sr in calibration source preparation

$$r_{Sr, Cal} = \frac{f_g \cdot (m_{oxal+pap, Cal} - m_{pap, Cal})}{C_{Sr, sol} \cdot m_{sol, Cal}} \quad \text{----- (3)}$$

f_g : gravimetric factor of Sr in monohydrate Sr-oxalate [$SrC_2O_4 \cdot H_2O$]

$m_{oxal+pap, Cal}$: total mass of Sr-oxalate and filter paper used in ^{90}Sr calibration source preparation [g]

$m_{pap, Cal}$: mass of filter paper used in ^{90}Sr calibration source preparation [g]

$C_{Sr, sol}$: concentration of Sr carrier solution [g Sr/g, solution]

$m_{sol, Cal}$: mass of Sr carrier solution taken for the calibration source preparation [g]

2.1.1. Uncertainty of chemical recovery of Sr

$$u_{r_{Sr, Cal}} = \sqrt{\left(\frac{r_{Sr, Cal}}{f_g}\right)^2 \cdot u_{f_g}^2 + \left(\frac{r_{Sr, Cal}}{(m_{oxal + pap, Cal} - m_{pap, Cal})}\right)^2 \cdot u_{m_{oxal + pap, Cal}}^2}$$

$$\sqrt{+ \left(\frac{r_{Sr, Cal}}{(m_{oxal + pap, Cal} - m_{pap, Cal})}\right)^2 \cdot u_{m_{pap, Cal}}^2 + \left(-\frac{r_{Sr}}{C_{Sr, sol}}\right)^2 \cdot u_{C_{Sr, sol}}^2}$$

$$\sqrt{+ \left(-\frac{r_{Sr}}{m_{sol, Cal}}\right)^2 \cdot u_{m_{sol, Cal}}^2}$$

----- (4)



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2.1.2. Uncertainties in weighing of sample, carrier and ^{90}Sr standard solution

$$m_i = m_{i+ti} - t_i \quad \text{-----} \quad (5)$$

$$u(t_i) = \frac{0.1}{\sqrt{3}} = 0.058 \text{ mg} \quad \text{-----} \quad (6)$$

$$u(m_i) = \sqrt{2 \times (0.058)^2} = 0.082 \text{ mg} \quad \text{-----} \quad (7)$$

m_i ; mass of material i

t_i ; uncertainty of weight of tare i

m_{i+ti} ; gross mass of material i
and tare i

2.1.3. Gravimetric factor and uncertainty

$$f_g = \frac{A_{Sr}}{M_{Sr-oxalate}} \quad \text{-----} \quad (8)$$

f_g : gravimetric factor

A_{Sr} : atomic mass of Sr [g, mol⁻¹]

$M_{Sr-oxalate}$: molecular weight of Sr-oxalate [g, mol⁻¹]

$$u_{f_g} = f_g \cdot \sqrt{\left\{ \left(\frac{u_{A_{Sr}}}{A_{Sr}} \right)^2 + \left(\frac{u_{M_{Sr-oxalate}}}{M_{Sr-oxalate}} \right)^2 \right\}} \quad \text{-----} \quad (9)$$

Table 1. Standard uncertainties of atomic weight and the gravimetric factor f_g (Delaeter, et. al., 2000)

| Element | Atomic weight, A , g mol ⁻¹ | Quoted uncertainty | Conversion factor | Standard uncertainty |
|---------|--|--------------------|-------------------|----------------------|
| Sr | 87.62 | 0.01 | $\sqrt{3}$ | 0.00577 |
| C | 12.0107 | 0.0008 | | 0.00046 |
| O | 15.9994 | 0.0003 | | 0.00017 |
| H | 1.00794 | 0.00007 | | 0.00004 |

| No. of atoms in a molecule of SrC ₂ O ₄ H ₂ O | Calculation g. mol ⁻¹ | Results g. mol ⁻¹ | Uncertainty | gravimetric factor | Uncertainty of the gravimetric factor, g.mol |
|--|----------------------------------|------------------------------|-------------|--------------------|--|
| Sr x 1 | 87.62 x 1 | 87.62 | 0.00577 | 0.45246 | 2.98 x 10 ⁻⁵ |
| C x 2 | 12.011 x 2 | 24.022 | 0.00092 | | |
| O x 5 | 15.9994 x 5 | 79.997 | 0.00085 | | |
| H x 2 | 1.00794 x 2 | 2.01588 | 0.00008 | | |
| Molecular weight, M | | 193.65488 | 0.00577 | | |



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Practical exercise 2 – uncertainty of chemical recovery

Please calculate **chemical recovery and its uncertainty** with the data given below, the uncertainty of the balance used is 0.1mg.

$$m_{\text{oxal+pap, Cal}} : 0.05566\text{g}$$

$$m_{\text{pap, Cal}} : 0.03562\text{ g}$$

$$C_{\text{Sr, sol}} : 0.00900 \pm 4.0 \times 10^{-7} \text{ g/g, sol.}$$

$$m_{\text{sol, Cal}} : \text{mass of empty vial} : 15.22134\text{ g}$$

$$\text{gross mass of empty vial and Sr carrier solution taken} : 16.32709\text{ g}$$

$$fg : 0.45246 \pm 0.0000298$$

Solution for Practical exercise 2

$$r_{Sr, Cal} = 0.45246 \cdot (0.05566 - 0.03562) / (0.00900 \cdot (16.32709 - 15.22134)) \\ = 0.911$$

$$u_{moxal+pap, Cal} = \frac{0.0001}{\sqrt{3}} = 0.000058 \text{ g}$$

$$u_{f_g} = 2.98 \times 10^{-5}$$

$$u_{pap, Cal} = \frac{0.0001}{\sqrt{3}} = 0.000058 \text{ g}$$

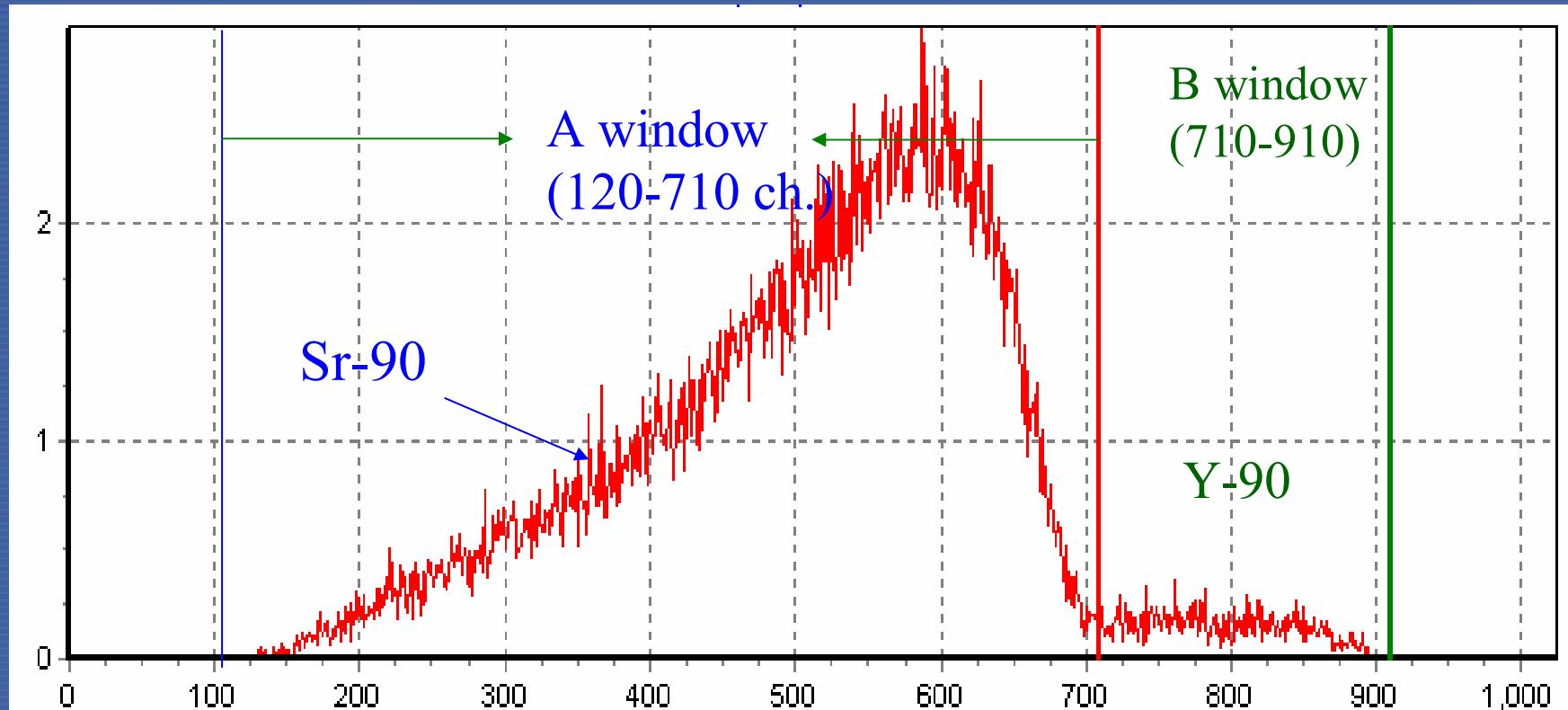
$$u_{m, Sol, Cal} = \sqrt{2 \times \left(\frac{0.0001}{\sqrt{3}}\right)^2} = 0.00008 \text{ g}$$

$$u_{CSr, Sol} = 4.0 \times 10^{-7} \text{ g, mol}^{-1}$$

$$u_{rSr} = 0.004$$

3. Determination of counting efficiency of ^{90}Sr

Spectra of Sr-90 calibration source



Net counts corrected in-growth Y-90 for Sr-90 = Net CPM in A win - f_y * Net CPM in B win

3.1. Statistics of count rates

$$K_{Cal} = I_{A, Cal} - I_{A, Bkg} - f_y \cdot (I_{B, Cal} - I_{B, Bkg}) \text{ ----- (10)}$$

$$u_{K_{Cal}} = \sqrt{u_{I_{A, Cal}}^2 + u_{I_{A, Bkg}}^2 + (I_{B, Cal} - I_{B, Bkg})^2 \cdot u_{f_y}^2 + f_y^2 (u_{I_{B, Cal}}^2 + u_{I_{B, Bkg}}^2)} \text{ ----- (11)}$$

K_{cal} : the net count rate corrected for in-growth of ^{90}Y in window A of ^{90}Sr calibration source spectrum [cpm]

$I_{A, Cal}$: gross count rate in window A of ^{90}Sr calibration source spectrum [cpm]

$I_{B, Cal}$: gross count rate in window B of ^{90}Sr calibration source spectrum [cpm]

3.1. Statistics of count rates

$$I_{A, Bkg} = \frac{1}{n} \sum_{i=1}^n I_{A, Bkg, i} \quad \text{-----} \quad (12)$$

$$u_{I_{A, Bkg}} = \sqrt{\frac{1}{n-1} \sum_{i=1}^n (I_{A, Bkg, i} - I_{A, Bkg})^2} \quad \text{-----} \quad (13)$$

3.2. Counting efficiency and its uncertainty

$$\varepsilon_{Sr} = \frac{K_{cal} \cdot \exp(-\lambda_{Sr} \cdot t_{1,cal})}{r_{Sr,cal} \cdot a_{0,Sr} \cdot 60} \quad \text{----- (14)}$$

$$u_{\varepsilon_{Sr}} = \sqrt{\left(\frac{\varepsilon_{Sr}}{K_{Cal}}\right)^2 \cdot u_{K_{Cal}}^2 + (\varepsilon_{Sr} \cdot t_{1,Cal})^2 \cdot u_{\lambda_{Sr}}^2 + \left(-\frac{\varepsilon_{Sr}}{a_{0,Sr}}\right)^2 \cdot u_{a_{0,Sr}}^2 + \left(-\frac{\varepsilon_{Sr}}{r_{Sr,Cal}}\right)^2 \cdot u_{r_{Sr,Cal}}^2} \quad \text{--- (15)}$$

K_{cal} : the net count rate corrected for in-growth of ^{90}Y in window A of ^{90}Sr calibration source spectrum [cpm]

$t_{1,cal}$: elapsed time between the reference date of ^{90}Sr standard and the separation time of Sr in the calibration source preparation [s]

λ_{Sr} : decay probability constant of ^{90}Sr [s^{-1}]

$r_{Sr,cal}$: recovery factor of Sr in the calibration source preparation

$a_{0,Sr}$: added activity concentration of ^{90}Sr certified standard at the reference date [Bq]

Practical exercise 3 – uncertainty of counting efficiency

- added activity concentration of ^{90}Sr certified standard at the reference date
 $a_{0,\text{Sr}} : 22.34 \pm 0.56 \text{ Bq/g}$
 - Mass of added ^{90}Sr standard solution ; $1.07776 \pm 8.16 \times 10^{-5} \text{ g}$
 - Half life of ^{90}Sr ; $28.9 \pm 0.03 \text{ year}$
 - Reference date of ^{90}Sr certified standard : 1991-01-01
 - Measurement time : 2007-05-15 14:26
- # it is assumed that the time interval between the separation date and measurement date, and the counting time are negligible compared to the half-life of Sr-90

| Sample name | replicate No. | Gross count rate (cpm) | | | |
|---------------|---------------|-------------------------------|-------|-------------------------------|-------|
| | | window A, $I_{A, \text{cal}}$ | u | window B, $I_{B, \text{cal}}$ | u |
| STD-1 | 1 | 1790.231 | 5.090 | 422.380 | 2.671 |
| | 2 | 1750.356 | 5.023 | 370.853 | 2.503 |
| | 3 | 1820.572 | 5.151 | 463.870 | 2.799 |
| Reagent blank | 1 | 6.936 | 0.187 | 2.346 | 0.109 |
| | 2 | 6.848 | 0.186 | 2.122 | 0.104 |
| | 3 | 6.789 | 0.185 | 2.052 | 0.102 |

Solution for Practical exercise 3

| Net count corrected for Y-90 in-growth, K_{Cal} | $u_{K_{\text{Cal}}}$ | Chemical recovery, $r_{\text{Sr, Cal}}$ | $U_{\text{Sr, Cal}}$ | λ_{Sr} | $u\lambda_{\text{Sr}}$ | $t_1, \text{Cal, (sec)}$ |
|--|----------------------|---|----------------------|-----------------------|------------------------|--------------------------|
| 1500.2 | 9.4 | 0.911 | 0.004 | 7.6E-10 | 7.89E-13 | 264047160 |
| 1495.0 | 9.2 | 0.911 | 0.004 | 7.6E-10 | 7.89E-13 | 264047160 |
| 1502.5 | 9.5 | 0.911 | 0.004 | 7.6E-10 | 7.89E-13 | 264047160 |

| Added ^{90}Sr activity | u | Counting efficiency, ϵ_{Sr} | $u\epsilon_{\text{Sr}}$ |
|---------------------------------|---------|---|-------------------------|
| 23.930 | 0.600 | 0.94 | 0.02 |
| 23.930 | 0.600 | 0.94 | 0.02 |
| 23.930 | 0.600 | 0.94 | 0.02 |
| | Average | 0.94 | 0.02 |

4. Sample preparation



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4.1. Ash/dry weight ratio and its uncertainty

$$f_{d/w} = \frac{(m_{dry+t1} - m_{t1})}{(m_{wet+t1} - m_{t1})} \quad (16)$$

$$f_{a/w} = \frac{(m_{ash+t2} - m_{t2})}{(m_{wet+t2} - m_{t2})} \quad (17)$$

$$f_{a/d} = \frac{f_{a/w}}{f_{d/w}} \quad (18)$$



$f_{d/w}$ = dry/wet ratio

$f_{a/w}$ = ash/wet ratio

$f_{a/d}$ = ash/dry ratio

m_{dry+t1} = gross mass of the dry subsample and the tare 1 [g]

m_{wet+t1} = gross mass of the wet subsample and the tare 1 [g]

m_{t1} = mass of tare 1 used for the dry/wet ratio determination [g]

m_{ash+t2} = gross mass of the ash subsample and the tare 2 [g]

m_{wet+t2} = gross mass of the wet subsample and the tare 2 [g]

m_{t2} = mass of tare t_2 used for the ash/wet ratio determination [g]



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$$u_c(f_{d/w}) = \sqrt{\frac{(u_{m_{dry+t1}}^2 + u_{m_{t1}}^2)}{(m_{dry+t1} - m_{t1})^2} + \frac{(u_{m_{wet+t1}}^2 + u_{m_{t1}}^2)}{(m_{wet+t1} - m_{t1})^2} - \left(\frac{2 \cdot u_{m_{t1}}^2}{(m_{dry+t1} - m_{t1}) \cdot (m_{wet+t1} - m_{t1})} \right)} \quad \text{----- (19)}$$

$$u_c(f_{a/w}) = \sqrt{\frac{(u_{m_{ash+t2}}^2 + u_{m_{t2}}^2)}{(m_{ash+t2} - m_{t2})^2} + \frac{(u_{m_{wet+t2}}^2 + u_{m_{t2}}^2)}{(m_{wet+t2} - m_{t2})^2} - \left(\frac{2 \cdot u_{m_{t2}}^2}{(m_{ash+t2} - m_{t2}) \cdot (m_{wet+t2} - m_{t2})} \right)} \quad \text{----- (20)}$$

$$u_c(f_{a/d}) = f_{a/d} \cdot \sqrt{\left(\left(\frac{uf_{a/w}}{f_{a/w}} \right)^2 + \left(\frac{uf_{d/w}}{f_{d/w}} \right)^2 \right)} \quad \text{----- (21)}$$

Practical exercise 4 – uncertainty of ash/dry wt ratio

We assume that
we used

- the balance associated with uncertainty of **0.1mg** to determine the dry/wet weight ratio
- **The weight of the tare 1** used for determining the dry/wet weight ratio is **32.66350 g**
- The gross mass of **tare 1 and wet sample** is **33.89030 g**
- The gross mass of **tare 1 and dry sample** is **33.87800 g**
- the balance with uncertainty of **0.1g** to determine the ash/wet weight ratio
- The **weight of the tare 2** used for determining the as/wet weight ratio is **728.32 g**
- The gross mass of **tare 2 and wet sample** is **1222.58 g**
- The gross mass of **tare 2 and ash sample** is **1197.94 g**

What are the ash/dry weight ratio and its uncertainty ?

Solution for Practical exercise 4

| | | |
|-----------------------------|---------------------------------|--------------------------------|
| MS of Tare 1(m_{t1}), g | Tare 1 + wet(m_{t1+wet}), g | Tare1 + dry(m_{t1+dry}), g |
| 32.6635 | 33.8903 | 33.878 |
| MS of Tare 2(m_{t2}), g | Tare 2 + wet(m_{t2+wet}), g | Tare2 + ash(m_{t2+ash}), g |
| 728.32 | 1222.58 | 1197.94 |

| | | | |
|-------------------------|------------|-------------------------|-------------|
| net wet(m_{wet}), g | U_{mwet} | net dry(m_{dry}), g | U_{mdry} |
| 1.2268 | 8.165E-05 | 1.2145 | 8.16497E-05 |
| net wet(m_{wet}), g | U_{mwet} | net ash(m_{ash}), g | U_{mash} |
| 494.26 | 0.08164966 | 469.62 | 0.081649658 |

| | |
|-----------|------------|
| $f_{d/w}$ | $U_{fd/w}$ |
| 0.9900 | 0.0001 |
| $f_{a/w}$ | $U_{fa/w}$ |
| 0.9501 | 0.0002 |
| $f_{a/d}$ | $U_{fa/d}$ |
| 0.9598 | 0.0002 |

5. Separation of Sr

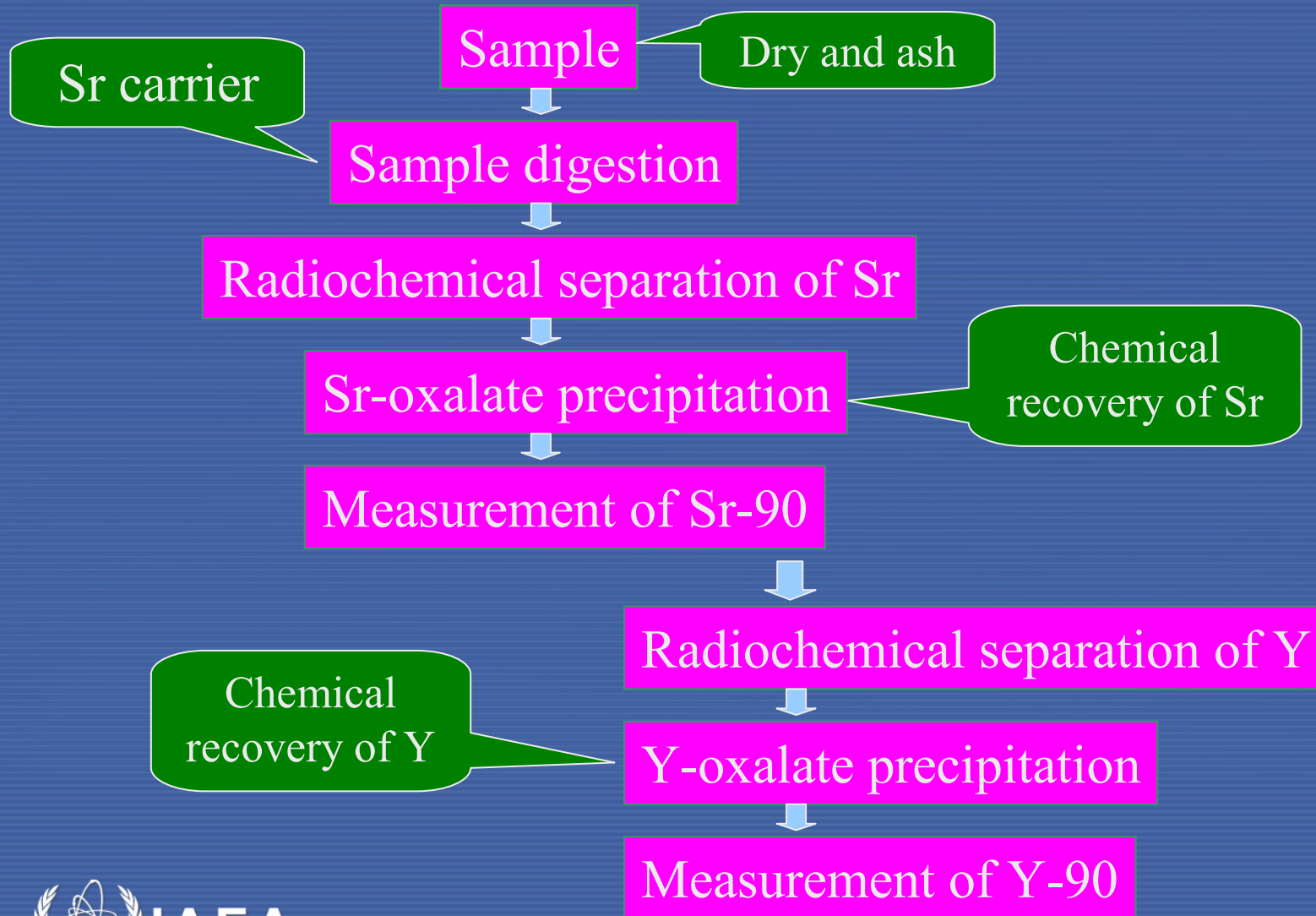


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Outline of the radiochemical procedure of Sr separation and source preparation



5.1. Chemical recovery of Sr from sample and uncertainty

$$r_{Sr} = \frac{f_g \cdot (m_{oxal+pap} - m_{pap})}{(C_{Sr, sol} \cdot m_{sol} + C_{Sr, nat} \cdot m_{ash})} \quad (22)$$

f_g : gravimetric factor of Sr in monohydrate Sr-oxalate ($SrC_2O_4 \cdot H_2O$)

$m_{oxal + pap}$: gross mass of Sr-oxalate and filter paper used for the source preparation from sample [g]

m_{pap} : mass of filter paper used for the source preparation from the sample [g]

$C_{Sr, sol}$: concentration of Sr carrier solution [g Sr/g, solution]

m_{sol} : mass of Sr carrier solution added to the sample [g]

$C_{Sr, nat}$: concentration of stable Sr in the sample [g Sr/g, ash]

m_{ash} : mass of ash sample [g]

Differentiating equation (21) and calculating the uncertainty of the chemical recovery,

$$u_{r_{Sr}} = \sqrt{\left(\frac{r_{Sr}}{f_g}\right)^2 \cdot u_{f_g}^2 + \left(\frac{r_{Sr}}{(m_{oxal+pap} - m_{pap})}\right)^2 \cdot u_{m_{oxal+pap}}^2 + \left(-\frac{r_{Sr}}{(m_{oxal+pap} - m_{pap})}\right)^2 \cdot u_{m_{pap}}^2}$$

$$\sqrt{\left(-\frac{r_{Sr} \cdot m_{sol}}{(C_{Sr,sol} \cdot m_{sol} + C_{Sr,nat} \cdot m_{ash})}\right)^2 \cdot u_{C_{Sr,sol}}^2 + \left(-\frac{r_{Sr} \cdot C_{Sr,sol}}{(C_{Sr,sol} \cdot m_{sol} + C_{Sr,nat} \cdot m_{ash})}\right)^2 \cdot u_{m_{sol}}^2}$$

$$\sqrt{\left(-\frac{r_{Sr} \cdot m_{ash}}{(C_{Sr,sol} \cdot m_{sol} + C_{Sr,nat} \cdot m_{ash})}\right)^2 \cdot u_{C_{Sr,nat}}^2 + \left(-\frac{r_{Sr} \cdot C_{Sr,nat}}{(C_{Sr,sol} \cdot m_{sol} + C_{Sr,nat} \cdot m_{ash})}\right)^2 \cdot u_{m_{ash}}^2}$$

----- (23)

Practical exercise 5 – uncertainty of chemical recovery in sample

Please calculate **chemical recovery and its uncertainty** with the data given below, the uncertainty of the balance used is 0.1mg.

$$m_{\text{oxal+pap}} : 0.05151 \text{ g}$$

$$m_{\text{pap, Cal}} : 0.03569 \text{ g}$$

$$C_{\text{Sr, sol}} : 0.00900 \pm 4.0 \times 10^{-7} \text{ g/g, sol.}$$

$$m_{\text{sol,}} : 1.12146 \pm 8 \times 10^{-5} \text{ g}$$

$$fg : 0.45246 \pm 0.00003$$

$$C_{\text{Sr, nal}} : 0.00011 \pm 0.00001 \text{ g Sr/g, ash}$$

$$m_{\text{ash}} : 7.05267 \pm 8 \times 10^{-5} \text{ g}$$

Solution for Practical exercise 5

| fg | ufg | $m_{\text{oxal+pap}}$ | $U_{\text{oxal+pap}}$ | m_{pap} | U_{pap} |
|---------|----------|-----------------------|-----------------------|------------------|------------------|
| 0.45246 | 2.98E-05 | 0.05151 | 0.000058 | 0.03569 | 0.000058 |

| $C_{\text{Sr, sol}}$ | $U_{\text{Sr, sol}}$ | m_{sol} | U_{mSol} | $C_{\text{Sr, Nat}}$ | $U_{\text{Sr, Nat}}$ |
|----------------------|----------------------|------------------|-------------------|----------------------|----------------------|
| 0.009 | 4.00E-07 | 1.12146 | 0.00008 | 0.00011 | 0.00001 |

| m_{ash} | U_{mash} | f_{Sr} | U_{rSr} |
|------------------|-------------------|-----------------|------------------|
| 7.05267 | 0.00008 | 0.659 | 0.005 |

6. Calculation of activity concentration of ^{90}Sr and its combined uncertainty on sampling date

6.1. Calculation of ^{90}Sr activity concentration on sampling date

$$a_{\text{Sr}} = \frac{[K \cdot f_{a/d} \cdot f_1 \cdot f_2] \times 1000}{m_{\text{ash}} \cdot \varepsilon_{\text{Sr}} \cdot r_{\text{Sr}} \cdot 60} \quad \text{-----} \quad (24)$$

a_{Sr} = activity concentration of ^{90}Sr in the sample on the sample collection date [Bq kg⁻¹, dry]

K = the net count rate corrected for in-growth of ^{90}Y in window A of ^{90}Sr spectrum from the sample [cpm]

$f_{a/d}$ = ratio of ash weight to dry weight

f_1 = correction factor for decay of ^{90}Sr during the time interval between the sampling date and the separation time of Sr

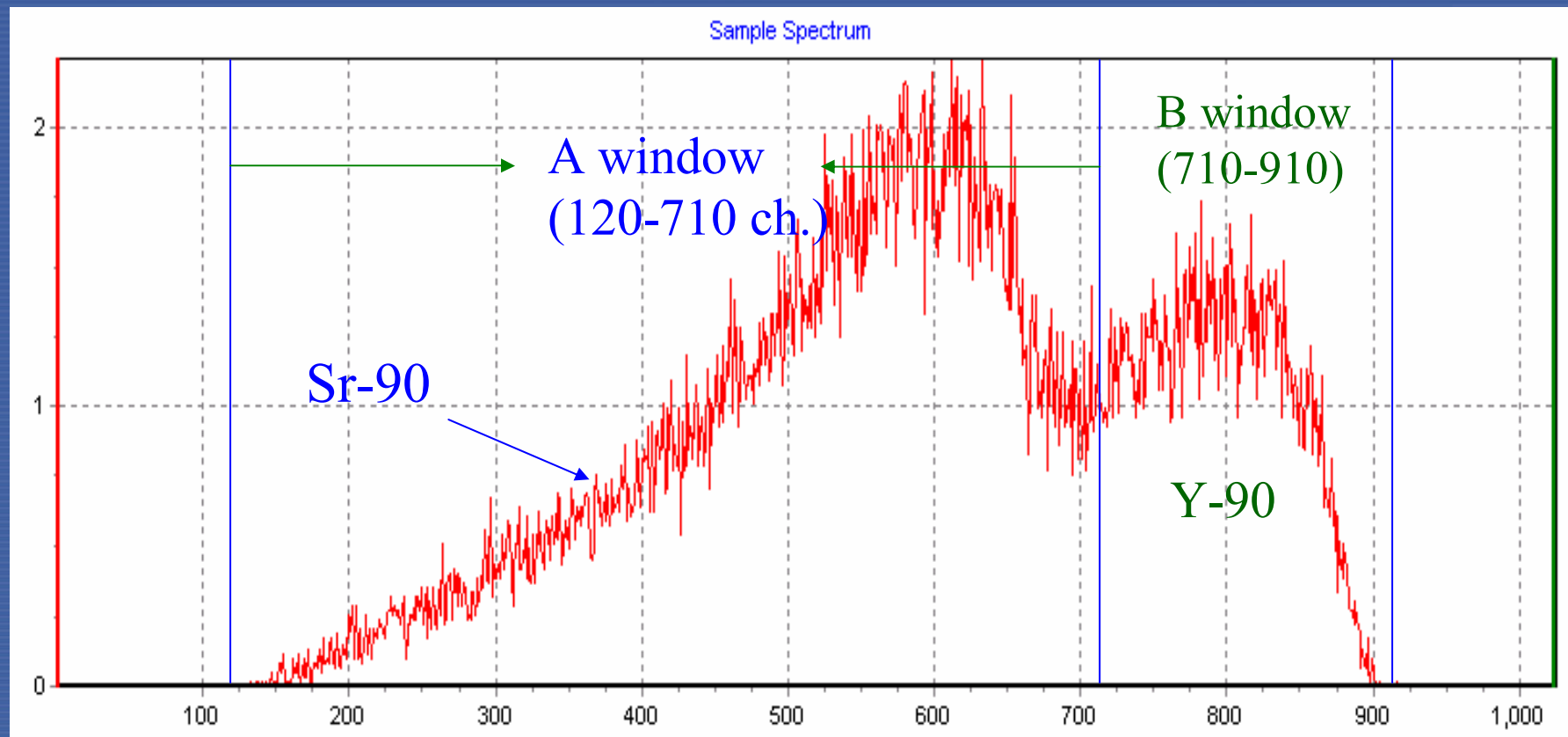
f_2 = correction factor for decay of ^{90}Sr during the time interval between the separation and the beginning of the measurement (** f_2 is negligible, if the sample is immediately measured after separation*)

ε_{Sr} = counting efficiency of ^{90}Sr

m_{ash} = mass of ash sample [kg]

r_{Sr} = chemical recovery factor of Sr in the sample

Spectrum of Sr-90 from sample



6.1.1. Net count rates in window A (Sr-90 region)

$$K = I_A - I_{A, BKG} - f_y \cdot (I_B - I_{B, BKG}) \quad \text{-----} \quad (25)$$

I_A = gross count rate in window A of ^{90}Sr spectrum from the sample [cpm]

I_B = gross count rate in window B of ^{90}Sr spectrum from the sample [cpm]

$I_{A, BKG}$ = count rate of blank in window A [cpm]

$I_{B, BKG}$ = count rate of blank in window B [cpm]

f_y = ratio of net count rates in the region A to that in the region B (“tailing factor”)

$$f_1 = \exp(\lambda_{\text{Sr}} \cdot t_1) \quad \text{-----} \quad (26)$$

λ_{Sr} = decay probability constant of ^{90}Sr [s^{-1}]

t_1 = the elapsed time between the sampling date and the separation time of ^{90}Sr [s^{-1}]

6.2. Calculation of combined uncertainty of ^{90}Sr activity on sampling date

$$u_c(a_{Sr}) = \sqrt{\left(\frac{a_{Sr}}{K}\right)^2 \cdot u_K^2 + \left(\frac{a_{Sr}}{f_{a/d}}\right)^2 \cdot u_{f_{a/d}}^2 + \left(-\frac{a_{Sr}}{m_{ash}}\right)^2 \cdot u_{m_{ash}}^2 + \left(-\frac{a_{Sr}}{\varepsilon_{Sr}}\right)^2 \cdot u_{\varepsilon_{Sr}}^2 + \left(-\frac{a_{Sr}}{r_{Sr}}\right)^2 \cdot u_{r_{Sr}}^2 + \left(\frac{a_{Sr}}{f_1}\right)^2 \cdot u_{f_1}^2 + \left(\frac{a_{Sr}}{f_2}\right)^2 \cdot u_{f_2}^2} \quad (27)$$

$$K = I_A - I_{A,Bkg} - f_y \cdot (I_B - I_{B,Bkg}) \quad (28)$$

$$u_K = \sqrt{u_{I_A}^2 + u_{I_{A,Bkg}}^2 + (I_B - I_{B,Bkg})^2 \cdot u_{f_y}^2 + f_y^2 (u_{I_B}^2 + u_{I_{B,Bkg}}^2)} \quad (29)$$

$$f_1 = \exp(\lambda_{Sr} \cdot t_1) \quad u_{f_1} = t_1 \cdot f_1 \cdot u_{\lambda_{Sr}} \quad (30)$$

Practical exercise 6 – ^{90}Sr activity and its combined uncertainty on sampling date

Please calculate ^{90}Sr activity and its combined uncertainty, if it assume that the measurement results of IAEA soil-6 are as follows;

it is assumed that the time interval between the separation date and measurement date, and the counting time are negligible compared to the half-life of Sr-90

| | | Gross cpm | | | |
|-------------|---------------|-----------------|----------|-----------------|----------|
| sample name | replicate No. | window A, I_A | U_{IA} | window B, I_B | U_{IB} |
| Soil-6 | 1 | 11.174 | 0.238 | 4.442 | 0.150 |
| | 2 | 10.895 | 0.235 | 4.402 | 0.149 |
| | 3 | 11.027 | 0.236 | 5.080 | 0.160 |
| Blank | 1 | 4.371 | 0.149 | 1.910 | 0.098 |
| | 2 | 4.295 | 0.148 | 1.580 | 0.089 |
| | 3 | 4.300 | 0.148 | 1.905 | 0.098 |

Solution for practical exercise 6

| | | Gross cpm | | | | | | Average | | | |
|-------------|---------------|--------------------------|-----------------|--------------------------|-----------------|-------------|---------------|-------------------------------|----------------------|-------------------------------|----------------------|
| sample name | replicate No. | window A, I _A | U _{IA} | window B, I _B | U _{IB} | sample name | replicate No. | window A, I _{A, Bkg} | U _{IA, Bkg} | window B, I _{B, Bkg} | U _{IB, Bkg} |
| Soil-6 | 1 | 11.174 | 0.238 | 4.442 | 0.150 | Soil-6 | 1 | | | | |
| | 2 | 10.895 | 0.235 | 4.402 | 0.149 | | 2 | | | | |
| | 3 | 11.027 | 0.236 | 5.080 | 0.160 | | 3 | | | | |
| Blank | 1 | 4.371 | 0.149 | 1.910 | 0.098 | Blank | 1 | | | | |
| | 2 | 4.295 | 0.148 | 1.580 | 0.089 | | 2 | | | | |
| | 3 | 4.300 | 0.148 | 1.905 | 0.098 | | 3 | 4.322 | 0.04248 | 1.798 | 0.18863 |

| f _y | U _{f_y} | Net count rate (cpm) corrected for ⁹⁰ Y in-growth | | f _{a/d} | U _{f_{a/d}} | m _{ash} | U _{mash} |
|----------------|----------------------------|--|----------------|------------------|------------------------------|------------------|-------------------|
| | | K | U _K | | | | |
| 0.674 | 0.008 | 5.07 | 0.29 | 0.9598 | 0.0002 | 7.05267 | 0.00008 |
| 0.674 | 0.008 | 4.82 | 0.29 | 0.9598 | 0.0002 | 7.05267 | 0.00008 |
| 0.674 | 0.008 | 4.49 | 0.29 | 0.9598 | 0.0002 | 7.05267 | 0.00008 |

| e _{Sr} | U _{e_{Sr}} | r _{Sr} | U _{r_{Sr}} | λ _{Sr} | Uλ _{Sr} | f _l | U _{f_l} | sampling date | separation date | t ₁ | a _{Sr} | U _{a_{Sr}} |
|-----------------|-----------------------------|-----------------|-----------------------------|-----------------|------------------|----------------|----------------------------|---------------|-----------------|----------------|-----------------|-----------------------------|
| 0.94 | 0.02 | 0.659 | 0.005 | 7.60E-10 | 7.89E-13 | 1.790543 | 1.08E-03 | 1983-01-30 | 2007-05-15 | 766454400.00 | 33.3 | 2.1 |
| 0.94 | 0.02 | 0.659 | 0.005 | 7.60E-10 | 7.89E-13 | 1.790543 | 1.08E-03 | 1983-01-30 | 2007-05-15 | 766454400.00 | 31.7 | 2.1 |
| 0.94 | 0.02 | 0.659 | 0.005 | 7.60E-10 | 7.89E-13 | 1.790543 | 1.08E-03 | 1983-01-30 | 2007-05-15 | 766454400.00 | 29.5 | 2.1 |
| | | | | | | | | | | Average | 31.5 | 2.1 |



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7. Preparation of spread sheet

Spreadsheet for calculation of combined uncertainty of ⁹⁰Sr in IAEA Soil-6

| Spreadsheet Method for Calculation Combined Uncertainty in the Determination of ⁹⁰ Sr in IAEA Soil-6 | | | | | | | | | | | | | | | | |
|---|--|-----------------|---------------|--------------|--------------|--------------|--------------|--------------|------------------|---------------------|-----------------|-----------------|---------------------------------|--------------|------------------|--|
| Sampling date | | | | | | | | | | Bq/g | | | | | | |
| 1983-01-30 | | | IA, cpm | IA, Bkg cpm | IB, cpm | IB, Bkg cpm | K, cpm | fy | f _{a/d} | m _{ash, g} | e _{Sr} | r _{Sr} | λ _{Sr, s⁻¹} | fl | t1, sec | |
| Separation date | Parameters | x _i | 11.174 | 4.322 | 4.442 | 1.798 | 5.070 | 0.674 | 9.60E-01 | 7.0527 | 0.940 | 0.659 | 7.6E-10 | 1.791 | 766454400 | |
| 2007-05-15 | Uncertainty | δx _i | 0.23800 | 4.248E-02 | 0.150 | 0.189 | 3E-01 | 0.0080 | 2.00E-04 | 0.0001 | 0.020 | 0.005 | 7.89E-13 | 1.08E-03 | 0 | |
| IA, cpm | 11.174 | | 11.412 | 11.174 | 11.174 | 11.174 | 11.174 | 11.174 | 11.174 | 11.174 | 11.174 | 11.174 | 11.174 | 11.174 | 11.174 | |
| IA, Bkg cpm | 4.322 | | 4.322 | 4.364 | 4.322 | 4.322 | 4.322 | 4.322 | 4.322 | 4.322 | 4.322 | 4.322 | 4.322 | 4.322 | 4.322 | |
| IB, cpm | 4.442 | | 4.442 | 4.442 | 4.592 | 4.442 | 4.442 | 4.442 | 4.442 | 4.442 | 4.442 | 4.442 | 4.442 | 4.442 | 4.442 | |
| IB, Bkg cpm | 1.798 | | 1.798 | 1.798 | 1.798 | 1.987 | 1.798 | 1.798 | 1.798 | 1.798 | 1.798 | 1.798 | 1.798 | 1.798 | 1.798 | |
| K, cpm | 5.070 | | 5.070 | 5.070 | 5.070 | 5.070 | 5.360 | 5.070 | 5.070 | 5.070 | 5.070 | 5.070 | 5.070 | 5.070 | 5.070 | |
| fy | 0.674 | | 0.674 | 0.674 | 0.674 | 0.674 | 0.674 | 0.682 | 0.674 | 0.674 | 0.674 | 0.674 | 0.674 | 0.674 | 0.674 | |
| f_{a/d} | 9.60E-01 | | 9.60E-01 | 9.60E-01 | 9.60E-01 | 9.60E-01 | 9.60E-01 | 9.60E-01 | 9.60E-01 | 9.60E-01 | 9.60E-01 | 9.60E-01 | 9.60E-01 | 9.60E-01 | 9.60E-01 | |
| m_{ash, g} | 7.0527 | | 7.0527 | 7.0527 | 7.0527 | 7.0527 | 7.0527 | 7.0527 | 7.0527 | 7.0528 | 7.0527 | 7.0527 | 7.0527 | 7.0527 | 7.0527 | |
| e_{Sr} | 0.940 | | 0.940 | 0.940 | 0.940 | 0.940 | 0.940 | 0.940 | 0.940 | 0.940 | 0.960 | 0.940 | 0.940 | 0.940 | 0.940 | |
| r_{Sr} | 0.659 | | 0.659 | 0.659 | 0.659 | 0.659 | 0.659 | 0.659 | 0.659 | 0.659 | 0.659 | 0.664 | 0.659 | 0.659 | 0.659 | |
| λ_{Sr, s⁻¹} | 7.6E-10 | | 7.6E-10 | 7.6E-10 | 7.6E-10 | 7.6E-10 | 7.6E-10 | 7.6E-10 | 7.6E-10 | 7.6E-10 | 7.6E-10 | 7.6E-10 | 7.6E-10 | 7.6E-10 | 7.6E-10 | |
| fl | 1.791 | | 1.791 | 1.791 | 1.791 | 1.791 | 1.791 | 1.791 | 1.791 | 1.791 | 1.791 | 1.791 | 1.791 | 1.792 | 1.791 | |
| t1, sec | 766454400 | | 766454400 | 766454400 | 766454400 | 766454400 | 766454400 | 766454400 | 766454400 | 766454400 | 766454400 | 766454400 | 766454400 | 766454400 | 766454400 | |
| a_{Sr, Bq/kg, dry} | 33.2 | | 34.8 | 33.0 | 32.6 | 34.1 | 33.2 | 33.1 | 33.2 | 33.2 | 32.5 | 33.0 | 33.2 | 33.3 | 33.2 | |
| a_i - a_{Sr} | | | 1.560 | -0.279 | -0.663 | 0.834 | 0.000 | -0.139 | 0.007 | 0.000 | -0.692 | -0.250 | 0.000 | 0.020 | 0.000 | |
| Σ(a_i - a_{Sr})² | 4.21E+00 | | 2.43E+00 | 7.76E-02 | 4.39E-01 | 6.95E-01 | 0.00E+00 | 1.92E-02 | 4.80E-05 | 1.42E-07 | 4.80E-01 | 6.26E-02 | 0.00E+00 | 4.02E-04 | 0.00E+00 | |
| U_{aSr, Bq/kg, dry} | 2.1 | | | | | | | | | | | | | | | |
| SF by Kragten | δa_{Sr}/δx_i | | 6.56E+00 | -6.56E+00 | -4.42E+00 | 4.42E+00 | 0.00E+00 | -1.73E+01 | 3.46E+01 | -4.71E+00 | -3.46E+01 | -5.01E+01 | 0.00E+00 | 1.86E+01 | 0.00E+00 | |
| Contribution, % | [(δa_{Sr}/δx_i)² · δx_i²] * 100 / U_{aSr}² | | 57.9 | 1.8 | 10.4 | 16.5 | 0.0 | 0.5 | 0.0 | 0.0 | 11.4 | 1.5 | 0.0 | 0.0 | 0.0 | |
| RSF, % | (δa_{Sr}/a_{Sr}) / (δx_i/x_i) | | 2.11 | -0.86 | -0.60 | 0.23 | 0.00 | -0.35 | 1.00 | -1.00 | -1.00 | -1.00 | 0.00 | 1.00 | 0.00 | |
| SF | Sensitivity Factor | | | | | | | | | | | | | | | |
| Contribution, % | Contribution of individual components to the combined uncertainty of the output quantity | | | | | | | | | | | | | | | |
| RSF, % | Relative Sensitivity Factor (relative change in the output quantity y divided by the relative change in the input quantity xi) | | | | | | | | | | | | | | | |

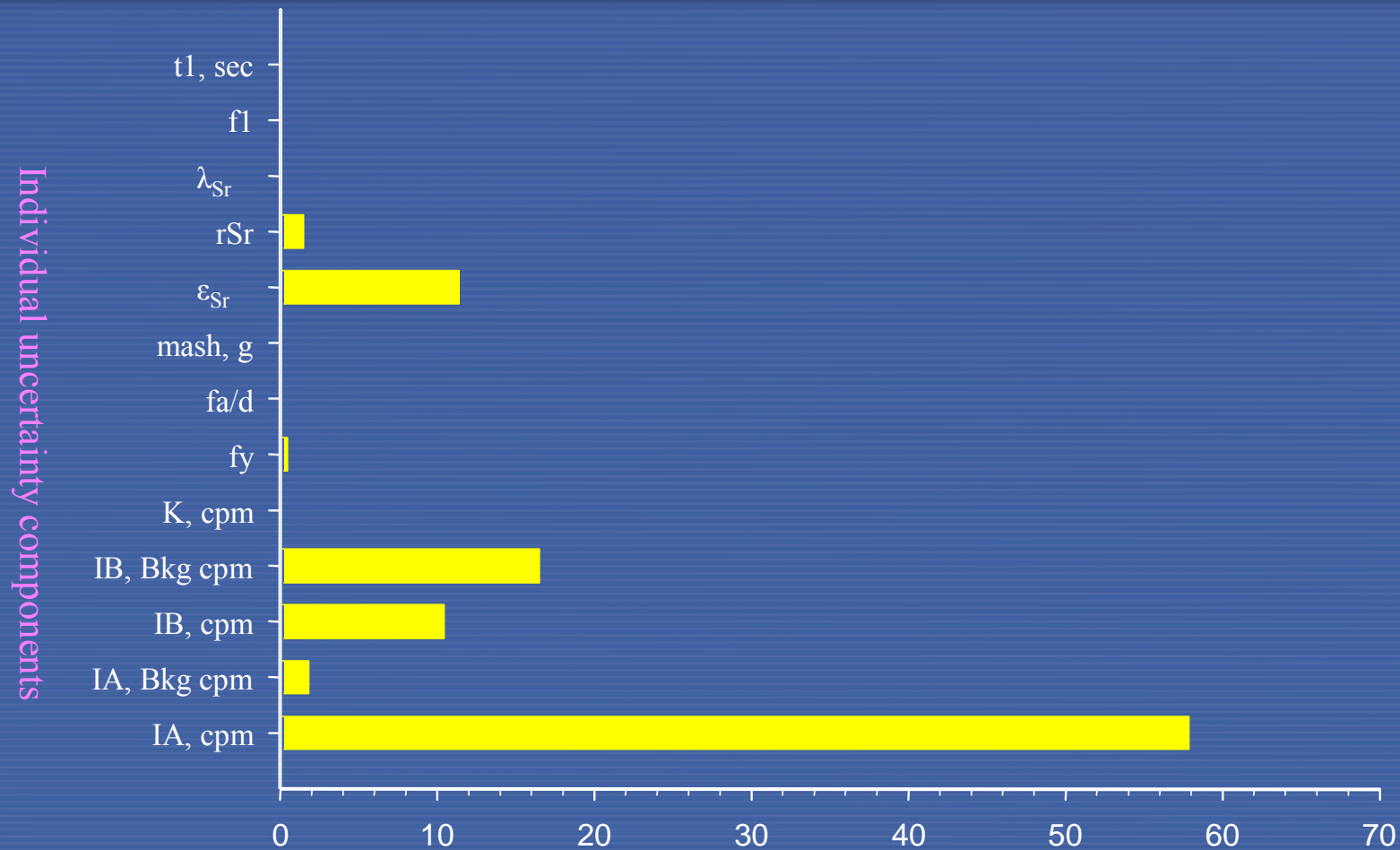


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Percentage contribution of individual uncertainty components to the combined uncertainty of ^{90}Sr in Soil-6



Percentage contribution of individual uncertainty components to the combined uncertainty(%)

Algebraic equations to calculate uncertainty

$$(A \pm u_A) \pm (B \pm u_B) = (A \pm B) \pm \sqrt{u_A^2 + u_B^2}$$

$$(A \pm u_A) \times (B \pm u_B) = (A \times B) \pm \sqrt{\left(\frac{u_A}{A}\right)^2 + \left(\frac{u_B}{B}\right)^2}$$

$$(A \pm u_A) \div (B \pm u_B) = (A \div B) \pm \sqrt{\left(\frac{u_A}{A}\right)^2 + \left(\frac{u_B}{B}\right)^2}$$

Calculation of Combined Uncertainty of ^{90}Sr activity using Algebraic Equation

| sample name | replicate No. | Gross cpm | | | | Average | | | | f_y | u_{f_y} | Net count rate (cpm) corrected for ^{90}Y in-growth | |
|-------------|---------------|-----------------|-----------|-----------------|-----------|-------------------------------|-------------------------|-------------------------------|-------------------------|-------|-----------|--|-------|
| | | window A, I_A | U_{I_A} | window B, I_B | U_{I_B} | window A, $I_{A, \text{Bkg}}$ | $U_{I_{A, \text{Bkg}}}$ | window B, $I_{B, \text{Bkg}}$ | $U_{I_{B, \text{Bkg}}}$ | | | K | U_K |
| Soil-6 | 1 | 11.174 | 0.238 | 4.442 | 0.150 | | | | | 0.674 | 0.008 | 5.07 | 0.29 |
| | 2 | 10.895 | 0.235 | 4.402 | 0.149 | | | | | 0.674 | 0.008 | 4.82 | 0.29 |
| | 3 | 11.027 | 0.236 | 5.080 | 0.160 | | | | | 0.674 | 0.008 | 4.49 | 0.29 |
| Blank | 1 | 4.371 | 0.149 | 1.910 | 0.098 | | | | | | | | |
| | 2 | 4.295 | 0.148 | 1.580 | 0.089 | | | | | | | | |
| | 3 | 4.300 | 0.148 | 1.905 | 0.098 | 4.322 | 0.0425 | 1.798 | 0.1886 | | | | |

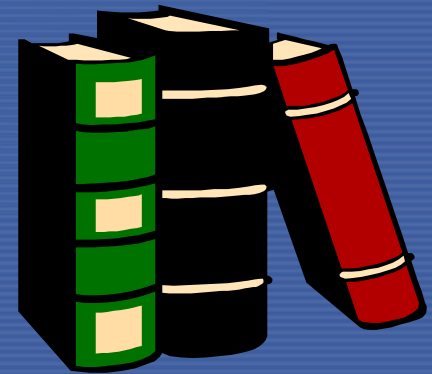
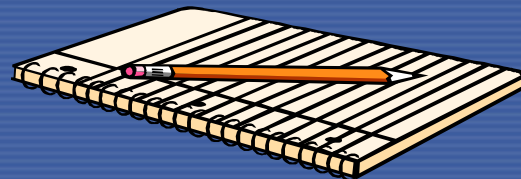
| $f_{a/d}$ | $u_{f_{a/d}}$ | m_{ash} | $u_{m_{\text{ash}}}$ | ϵ_{Sr} | $u_{\epsilon_{\text{Sr}}}$ | r_{Sr} | $u_{r_{\text{Sr}}}$ | λ_{Sr} | $u_{\lambda_{\text{Sr}}}$ | f1 | u_{f1} |
|-----------|---------------|------------------|----------------------|------------------------|----------------------------|-----------------|---------------------|-----------------------|---------------------------|----------|----------|
| 0.9598 | 0.0002 | 7.05267 | 0.00008 | 0.94 | 0.02 | 0.659 | 0.005 | 7.60E-10 | 7.89E-13 | 1.790519 | 1.08E-03 |
| 0.9598 | 0.0002 | 7.05267 | 0.00008 | 0.94 | 0.02 | 0.659 | 0.005 | 7.60E-10 | 7.89E-13 | 1.790543 | 1.08E-03 |
| 0.9598 | 0.0002 | 7.05267 | 0.00008 | 0.94 | 0.02 | 0.659 | 0.005 | 7.60E-10 | 7.89E-13 | 1.790543 | 1.08E-03 |

Calculation of Combined Uncertainty of ^{90}Sr activity using Algebraic Equation

| sampling date | separation date | t_1 | a_{Sr} | $U_{a\text{Sr}}$ |
|---------------|-----------------|--------------|-----------------|------------------|
| 1983-01-30 | 2007-05-15 | 766454400.00 | 33.2 | 2.1 |
| 1983-01-30 | 2007-05-15 | 766454400.00 | 31.7 | 2.1 |
| 1983-01-30 | 2007-05-15 | 766454400.00 | 29.5 | 2.1 |
| | | Average | 31.5 | 2.1 |

Good Sources of Information

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Thank you for your attention



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