VALIDATION OF ORGANICALLY BOND TRITIUM AND C-14 ANALYSIS ON ENVIRONMENTAL SAMPLES

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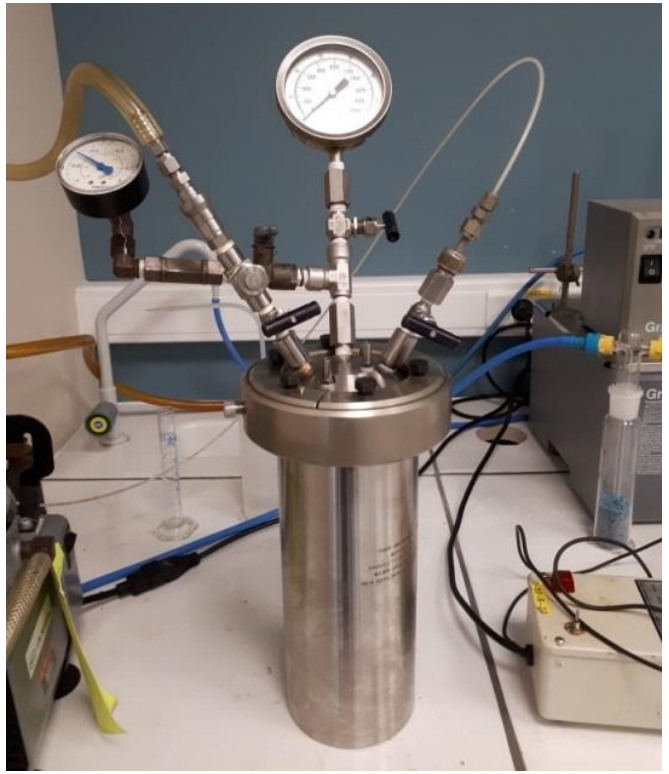


Parr bomb

Introduction

2 entities, 3 business lines

~ 250 people
~ 100 M€

Our priority?

Promoting the beneficial use of radioisotopes for medical purposes whilst ensuring the absence of harmfulness

Management team

Public Utility Foundation
founded in **1971**

Limited company
founded in **2010**

Analysis and control of radioactivity

Radioisotopes

Radiopharmaceuticals

IRE Lab, keeping radiation safe

- IRE's preventive branch, **IRE Lab** contributes to protecting the environment, workers and the population

- Through its personalized support and recognized industrial experience, **IRE Lab** is the reference partner for global consultancy and customized solutions for the **analysis and monitoring of radioactivity**

IRE Lab
Keeping radiation safe

IRE | IRE Elit Radiopharma

A large range of samples

~ 8500 analyses per year

1 **Environmental monitoring**
Groundwater, surface water, rain water, soil, sediment, dust, aerosol, flora and fauna

2 **Monitoring of water for human consumption**
Tap water, spring water, mineral water, drinks

3 **Control of food chain**
Foodstuffs, agricultural products, crops and livestock products

4 **Monitoring of workers**
In vitro (urine, faeces, nasal mucus) or in vivo (whole body, thyroid)

5 **Support for industrial, nuclear and non-nuclear sectors**
Dismantling of nuclear facilities, radioactive waste, NORM, industrial waste, waste water, construction material

6 **Checks on goods for import and export and various consumer goods**

IRE | IRE Elit Radiopharma

OBT and C-14 analysis in routine

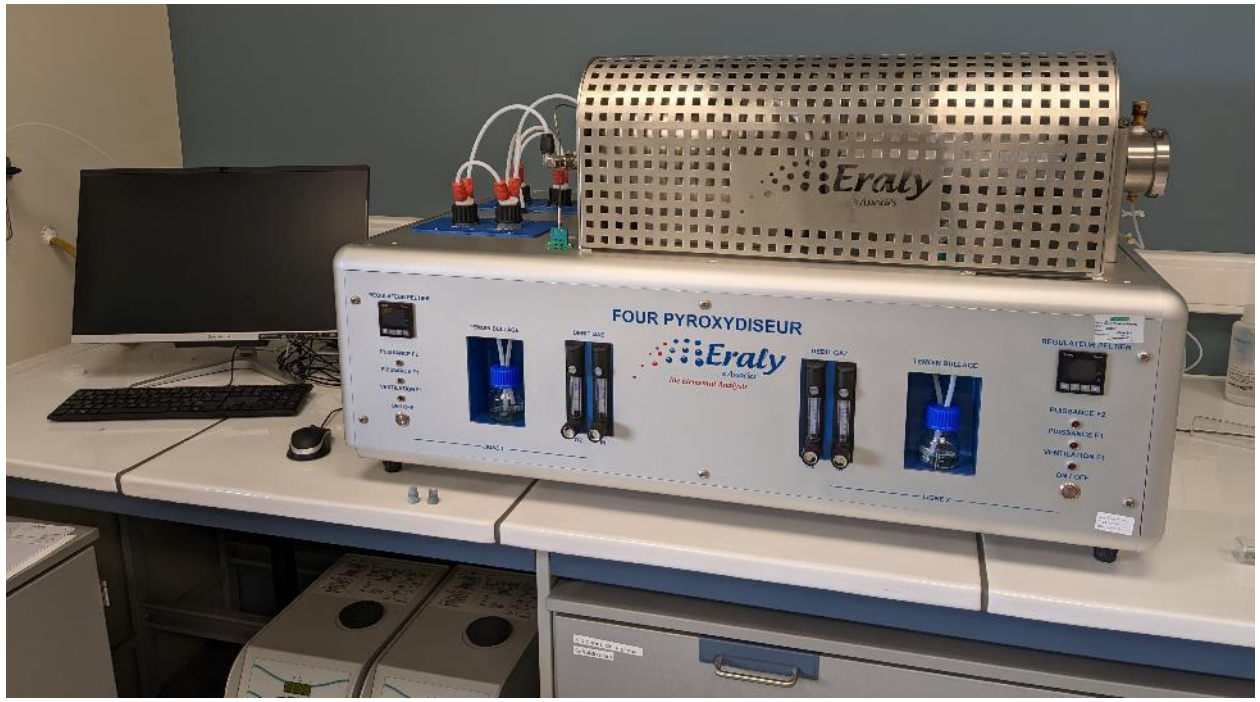
- Within the framework of the Belgian radiological monitoring program
- Food chain : C-14
- Aquatic fauna and flora from the river Meuse : OBT
- Agricultural samples around the NPP Chooz and Tihange : OBT and C-14
- Total ~ 90 – 100 samples per year

Nuclear power plant of Chooz

Materials and analytical method for OBT and C-14 determination

The Eraly Pyroxydizer furnace

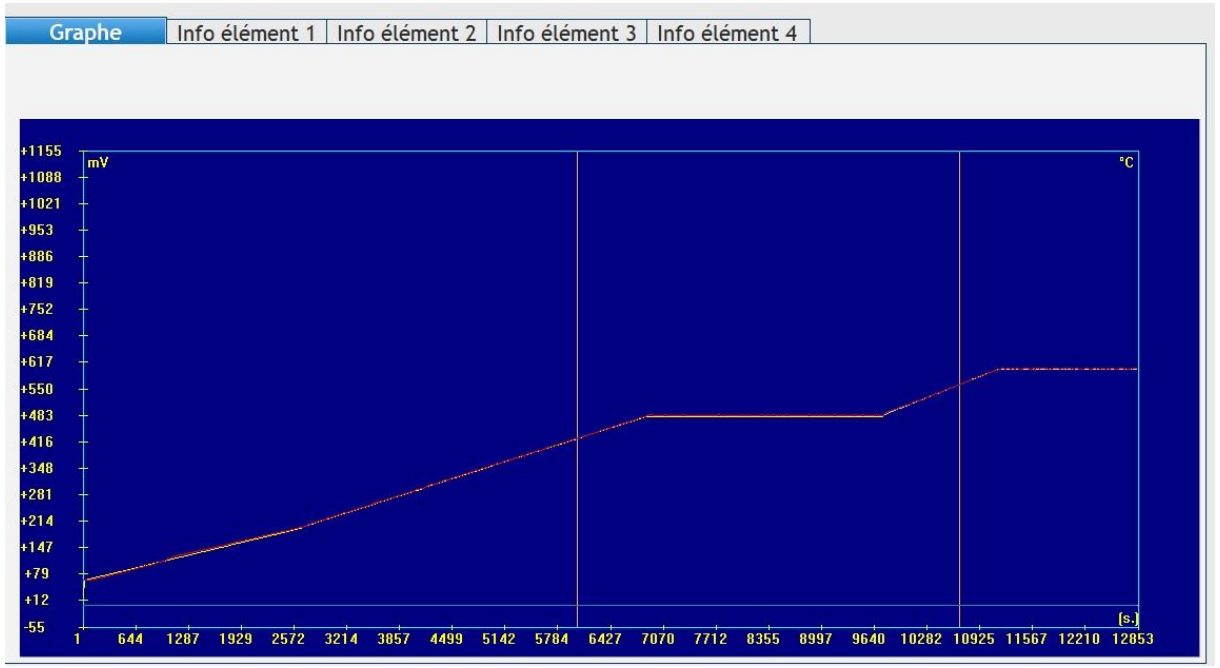
- 2 independant combustion lignes (1 to 3)
- Two differents zones at high temperatures
  - Pyrolysis (70°C - 950°C)
  - Oxydation (800°C – 1000°C)
- Two cooling traps (up to -20°C)
- Required Oxygene and inert gas inlets



Pyroxydizer furnace® (Eraly & Associés)

Helios software

Each line has its dedicated software



Temperature ramp for plant analysis

Analys methodology

plants → Drying at 40°c and grinding → Sample quantity 5g → OBT / C-14 extraction

OBT → 1 cooling trap with 30 ml of dead water → 2 x (9:10) vials with Quicksafe 450 cocktail

C-14 → 2 cooling traps with 30 ml Oxysolve C-400 LSC → 2 x 18 ml LSC vials

LSC measurements on TriCarb 3180TR (Perkin Elmer). No quenching corr. (spike/non spike)

Analytical performances

OBT (Bq/kg d.w.)

- Bkg = 1.20 cpm
- Counting time = 2 x 100 minutes
- Counting eff. = 20%
- Combustion eff. = 97%
- LoQ ~ 20 Bq/kg

C-14 (Bq/g carbon)

- Bkg = 1.80 cpm
- Counting time = 2 x 100 minutes
- Counting eff. = 70%
- Combustion eff. = 97%
- LoQ ~ 0.1 Bq/g C

Validation plan

Validation plan according ISO 17025 standard

- Method based on NF M60-824 standard - Test method for the analysis of tritium in free water and organically bound tritium in environmental matrices
- OBT : Standard method = verification / C-14 : Internal method = validation
- Environmental matrices
- Validation tests for OBT:
  - Trueness
  - Repeatability
  - Limit of Detection
  - Other : Memory effects, FoM, delay time

444-TEST

Results of the validation tests

Trueness

- Using reference materials from previous proficiency tests of 2013 and 2020
- Evalution is performed with the Zeta-score [-2;+2] and the Bias [<15%]

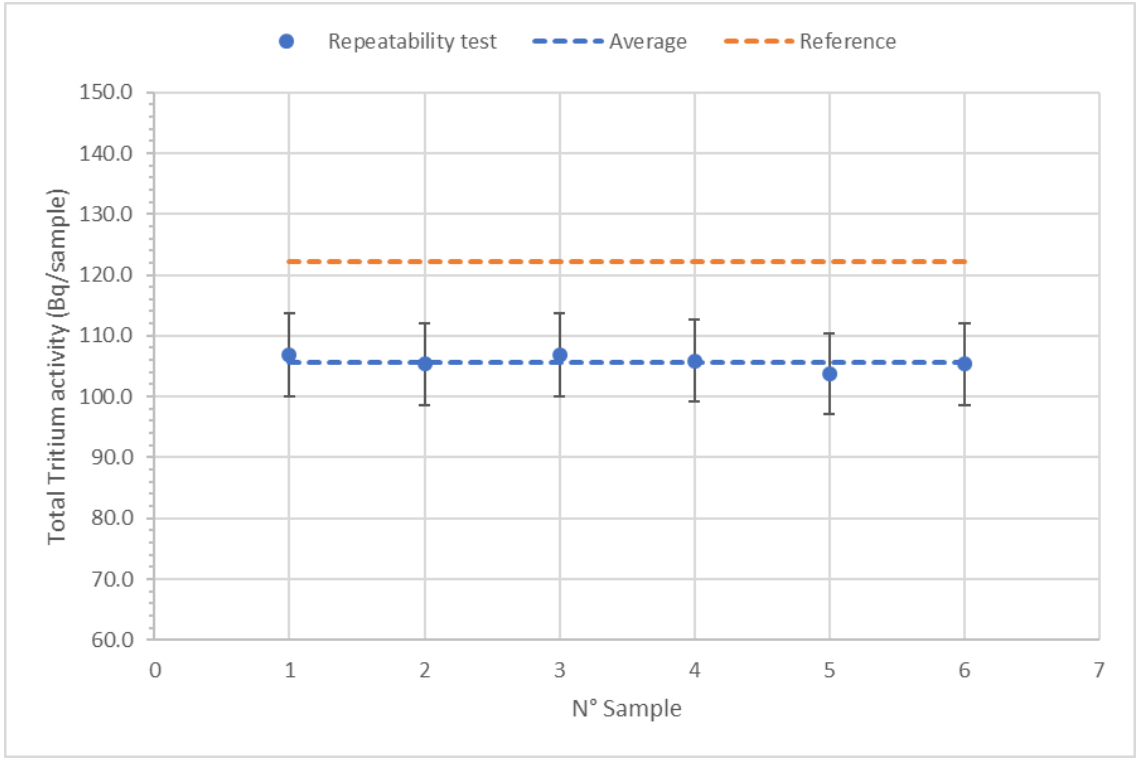
Sample ID	Parameter	Ref. act.	unc. Ref (k=1)	Results	unc. Results (k=1)	unit	Zeta-score	Bias
IRSN 171V300 - ligne 1	OBT	94	7	87	8	Bq/kg d.w.	**-0.7**	**-7.9%**
IRSN 171V300 - ligne 2	OBT	94	7	81	8	Bq/kg d.w.	**-1.2**	**-13.8%**

- C-14

Sample ID	Parameter	Ref. act.	unc. Ref (k=1)	Results	unc. Results (k=1)	unit	Zeta-score	Bias
IRSN 127V300 - ligne 1	C-14	468	12.5	492	18.45	Bq/kg C	**1.1**	**5.1%**

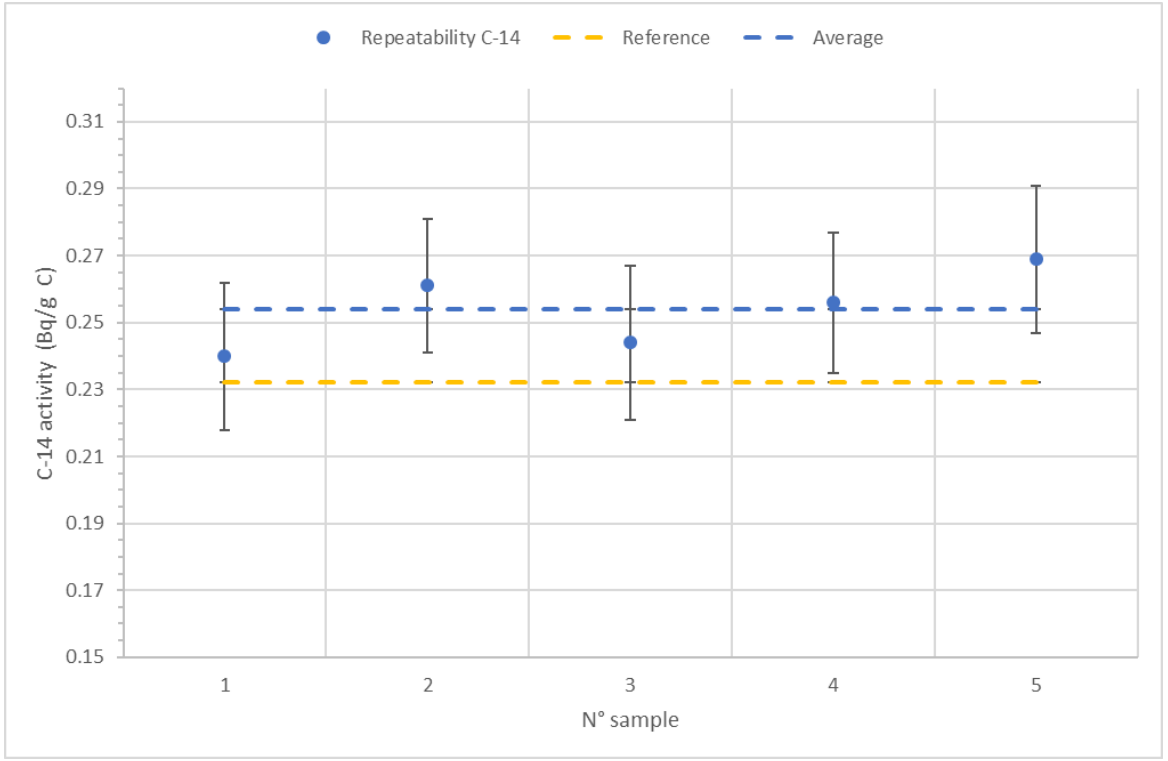
Repeatability test (H-3)

- Sample = 5 g of plants spiked with ~120 Bq of H-3 (HTO)
- H-3 = 20 kBq/kg d.w.
- RSD = 1.1%
- Memory effect? No trend
- But 13% negative bias compare with the reference H-3 activity
- Negative bias is under investigation
  - Redo the test with 2 cold traps



Repeatability test (C-14)

- Sample = 5 g of plants (willow leaf)
- « C-14 reference activity » = 0.232 Bq/g C*
- No C-14 spike
- RSD = 4.7%
- Memory effect? No trend
- Bias = 9.5%
- Ratio g C/g Samle (d.w.) = 0.32



** Roussel-Debet S., Radioprotection 49(1), 49-54 (2014)*

Conclusions

What's next - future developments

- Investigation of negative bias of OBT analyses

  Determination of tritium loss with a second trap

- Experimental determination of the recovery factor and its related uncertainty

  Using a glucose solution of known concentration for C-14?

- Improved carbon recovery rate

  Investigate others liquids scintillation cocktail?

- Need for certified reference materials and proficiency tests!

- Your advices and feed-backs are welcome!

Thank you for your attention

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