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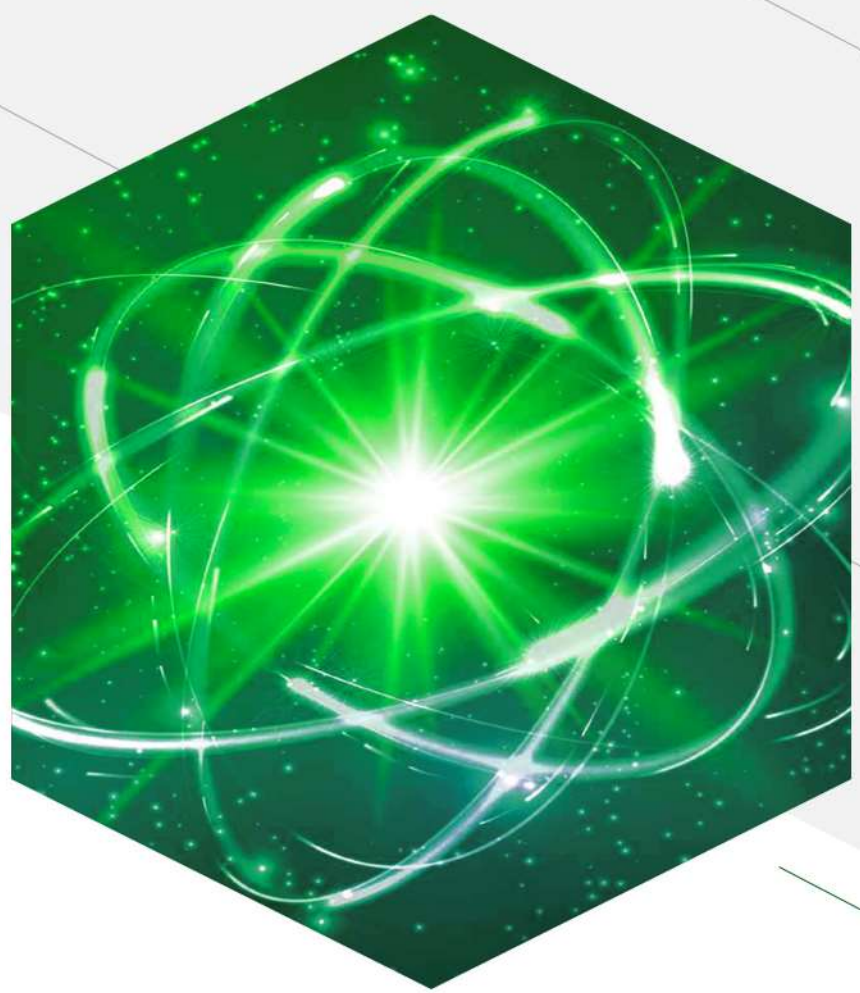

Neutron Activation Analysis with Short-Half-Life Radionuclides: Challenges, Opportunities, and Implementation Guidelines

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Technical Meeting on Neutron-induced Reactions on Short-lived Nuclei, 25 to 29 August 2025, Vienna

Outline

- Challenges & limitations in NAA
- Opportunities with SHL Radionuclides
- Classification of Radionuclides by Half-Life
- Implementing NAA with SHL Radionuclides
- NAA with SHL at the Moroccan Triga Mark II Research Reactor

Challenges & limitations in NAA

- Most widely applied analytical technique at research reactors.
- Typical procedure:
- One or more irradiations
- Successive measurements after decay periods (up to 3 - 4 weeks).
- Such Long decay periods lead to long sample turnaround times → less competitive vs. alternative analytical techniques (ICP-MS, AAS, XRF…..).

Challenges & limitations in NAA

Another limitation to NAA is the small, low-flux reactors in such laboratories :

- **Limited capacity (few samples can be irradiated simultaneously).**
- **Low induced activity may require relatively long irradiation times and counting times, thus limiting the number of samples that can be processed.**

- **NAA laboratories are searching for ways to anticipate on such limitations and for new niches as a service provider**
- **During the (virtual) Training Workshop on Optimization of Performance and Processes in Neutron Activation Analysis that took place in 2020 recommended to the IAEA to consider developing guidance and practical advice on all aspects of NAA with short half-life radionuclides**

Opportunities with SHL Radionuclides

- Radionuclides with short half-lives, from seconds to minutes and a few hours, are worth to be explored for their potential.

- Some laboratories already measure a few of such short half-life radionuclides in their routine schemes, e.g., for the assessment of Al, Mg, Mn, Ti, V, Cu and Ca.

- The number of elements that can be measured by such short half-life radionuclides is much larger and encompasses several that can widen the interest for NAA, such as F, Se, I, Cl, Br and even Li.

Opportunities with SHL Radionuclides

- It is obvious that such measurements with durations of minutes or even seconds immediately provide an outlook for reporting results very quickly, even on the same day of the analysis, and also to increase considerably the competitiveness of NAA as well as the analysis throughput.

- This opportunity is not fully employed by NAA laboratories because of many technical and analytical difficulties. Overcoming these issues and implementing NAA with short half-life radionuclides can provide labs with new opportunities and improve their competitiveness in the market for multi-elemental non-destructive analytical techniques.

Classification of Radionuclides by Half-Life

- The radionuclides produced by neutron irradiation may have half-lives varying from a fraction of a second to days, or even years. There is no formal convention for the classification of radionuclides on the basis of the length of their half-lives.

- **Commonly, radionuclides with half-lives varying from approximately a few seconds to approximately two hours are, within the NAA community, considered to be the 'short half-life' radionuclides (or ‘short-lived’, in NAA jargon); radionuclides with half-lives shorter than about one minute are sometimes classified as ‘ultra shorts’.**

Classification of Radionuclides by Half-Life

- There is no common annotation for the group of radionuclides with half-lives up to approximately two days. These are referred to as ‘medium half-life radionuclides, whereas radionuclides with longer half-lives are considered to be ‘long half-life’ radionuclides.

- NAA with short half-life radionuclides enables analysis turnaround time of, in principle, one working day.

- It has been applied at research reactors, neutron generators and isotopic neutron sources ever since the development of NAA.

Implementing NAA with SHL Radionuclides

- To successfully use short half-life radionuclides in Neutron Activation Analysis (NAA), it is essential to evaluate and possibly modify the entire analytical process. This includes the irradiation facility, the counting equipment, and the calibration approach.

- This presentation provides comprehensive guidance on implementing and optimizing Neutron Activation Analysis (NAA) with short half-life radionuclides. It covers all relevant aspects for research reactors, neutron generators, and isotopic neutron sources.

Implementing NAA with SHL Radionuclides

- It focuses primarily on the experimental details and sensitivity of research reactor-based Instrumental NAA (INAA), it does not address Radiochemical NAA (RNAA) with short half-life radionuclides.

- All these topics have been discussed and reported during the (virtual)

“Consultancy Meeting on the Development of a Publication on Neutron Activation Analysis with Short Half-Life Radioisotope, 13-15 April 2021”

NAA at the Moroccan Triga Mark II Research Reactor

Power : 2 MW

(Start-up : Mai 2007)

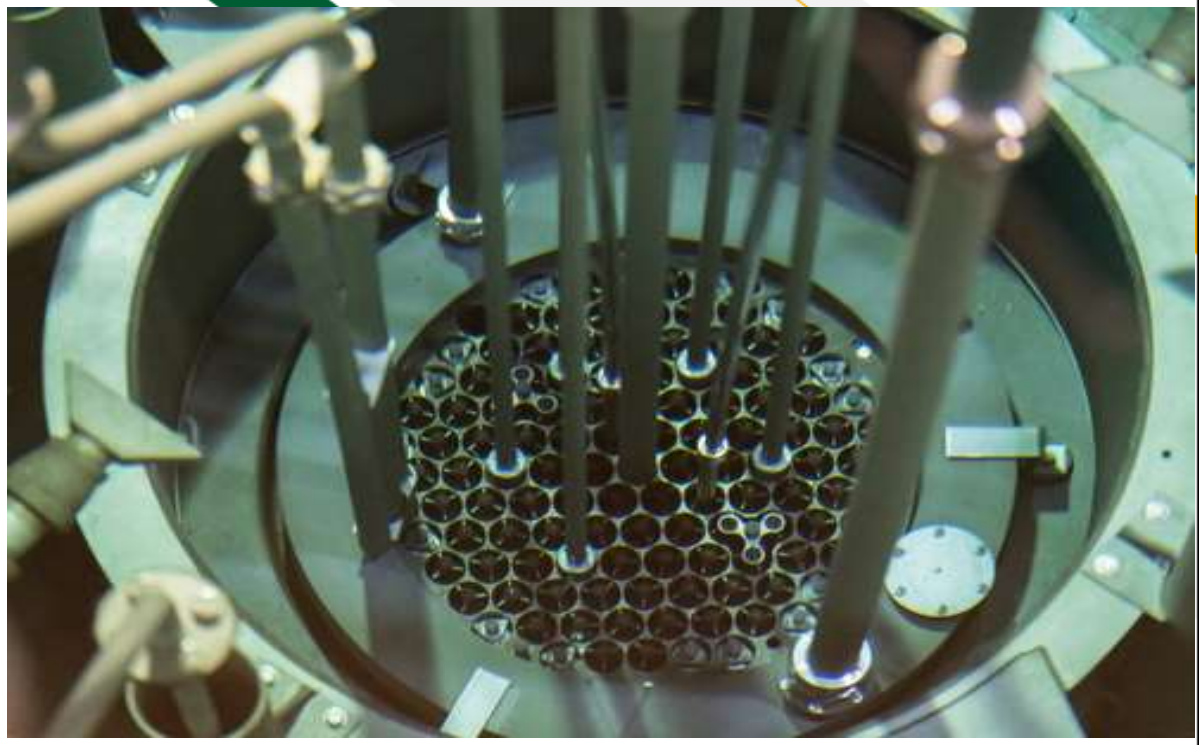
4 beam ports : 3 radials et 01 tangential

+ thermal column

(INAA) was introduced by **comparative Method** since 2010.

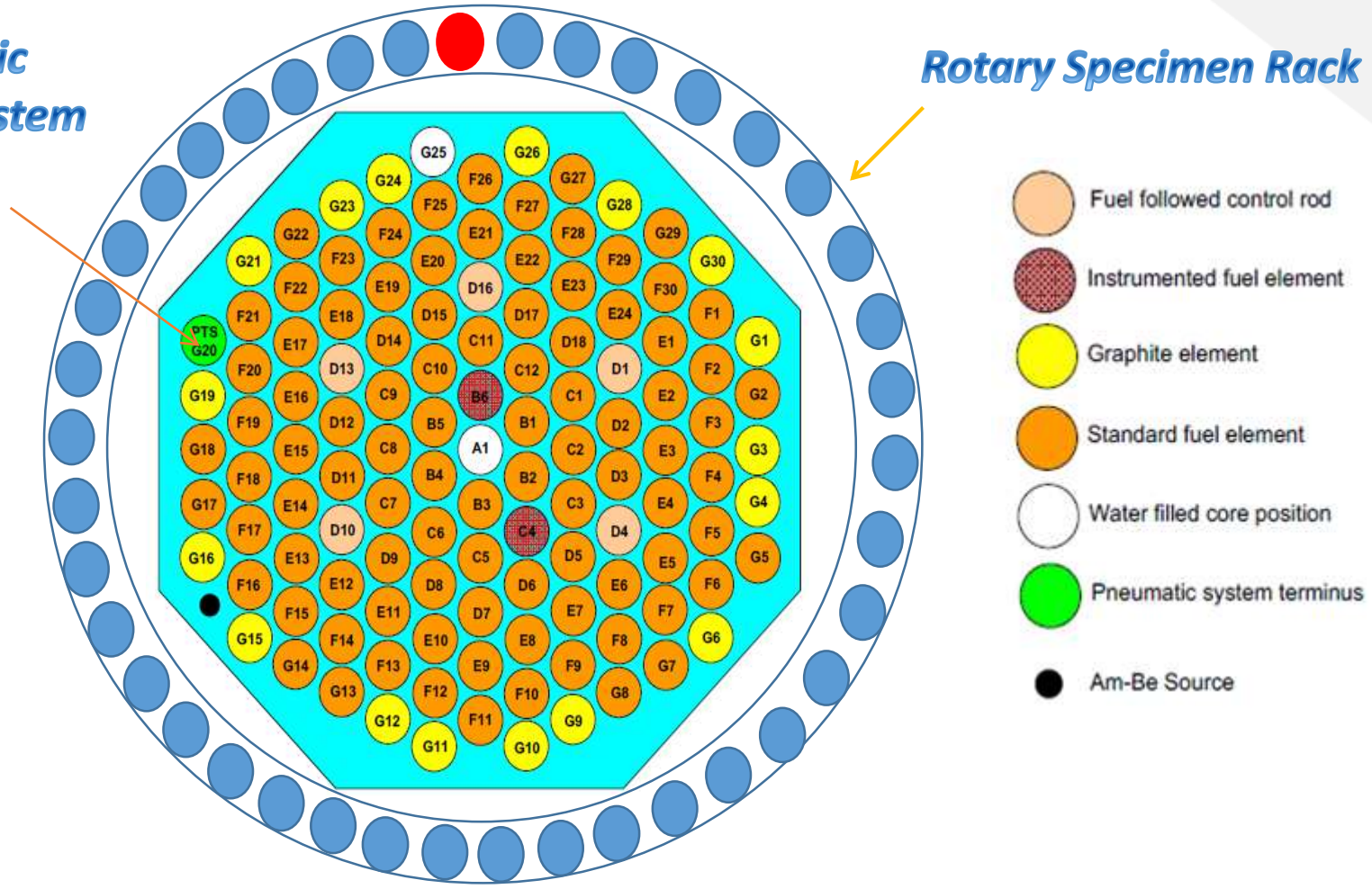
k0-INAA method was started in June 2013 by a cooperation project with reactor institute delft (RID)

NAA at the Moroccan Triga Mark II Research Reactor



- Neutron Activation Analysis
- Isotope production
- Neutron Imaging (in progress)
- Neutron Diffraction (in progress)

NAA Irradiation positions

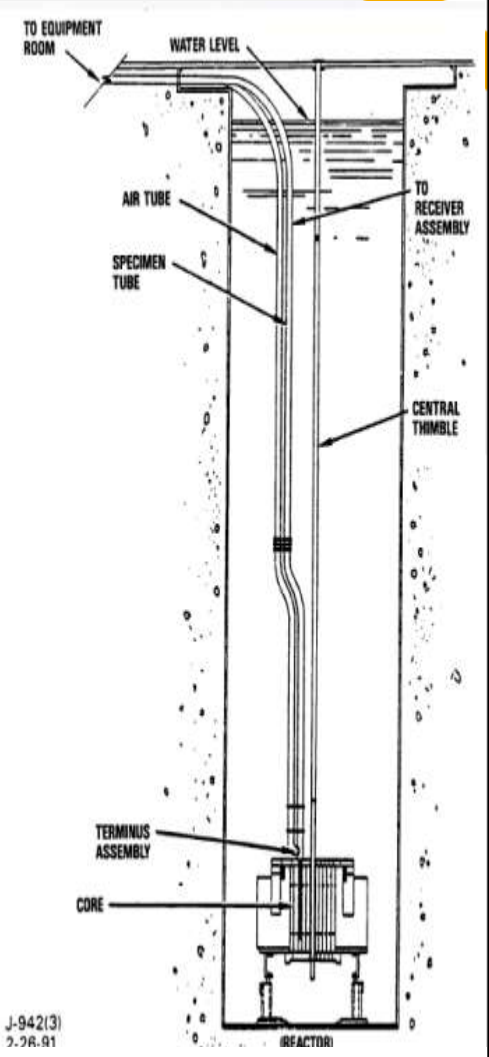


Pneumatic transfer system in-core terminus

Pneumatic transfer system, used for irradiation of short-lived radioisotopes.

The in-core terminus of this system is normally located in the outer ring of fuel element positions, a region of high neutron flux.

The sample capsule (rabbit) is conveyed to a receiver-sender station via 3.18 cm o.d. aluminium tubing.



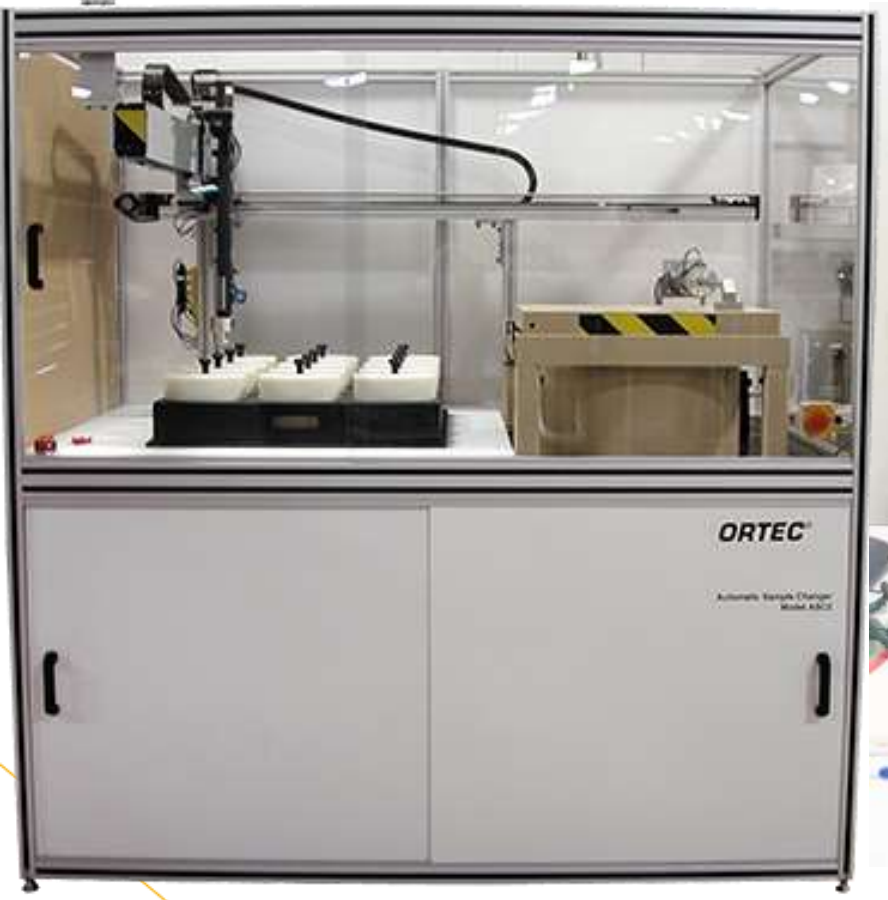
Irradiation protocoles for short irradiations

Φ_{th}=8.5 10 ^{11} n.cm^{-2}.sec^{-1}

Rabbits have been transferred via pneumatic transfer system to the reactor core from NAA laboratory.

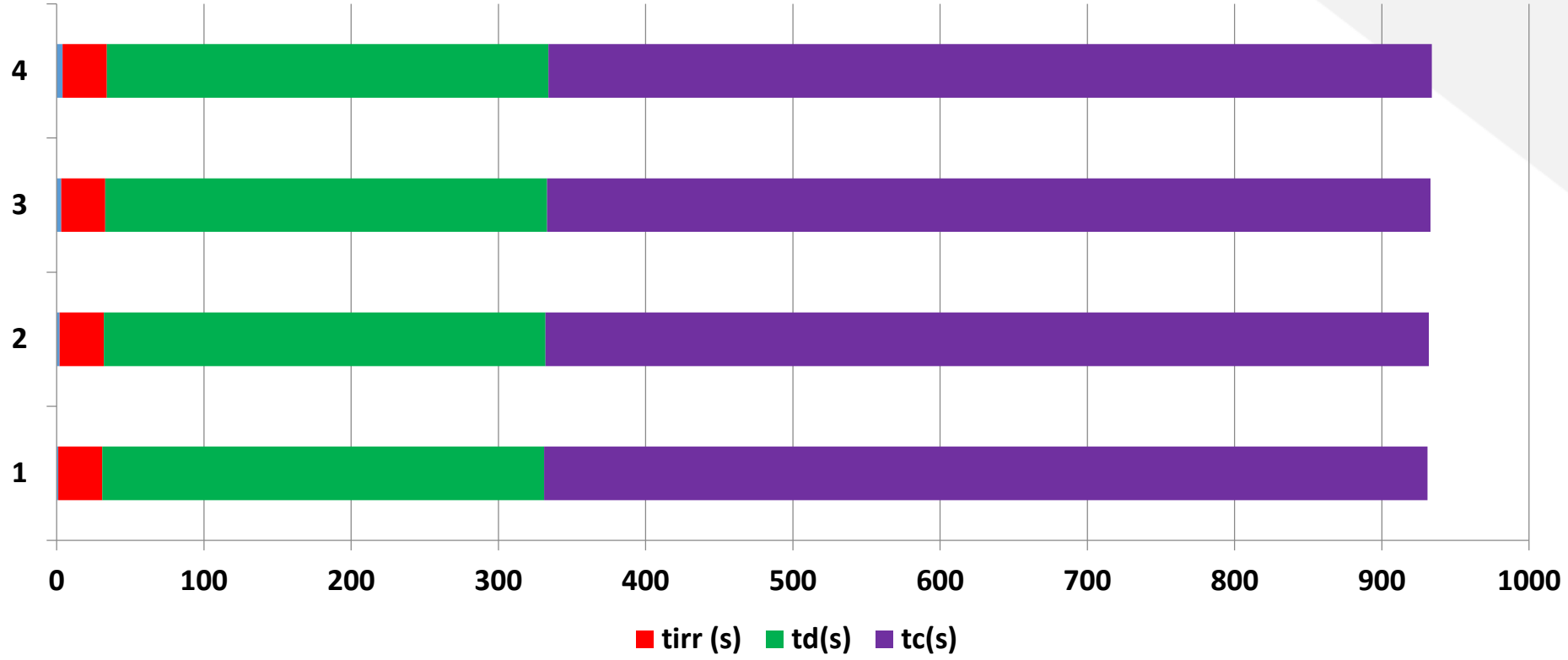
Each rabbit have been irradiated alone for 30 s with Al-0,1%Au used as flux monitor.

PTS



03 Vertical HPGe P-type Detector with 30% Relative efficiency (FWHM=1.85 keV)

Things we learned: Overlapping irradiation, decay and counting: Al, Mg, Mn, V, Ti, Ca, Cl….



Irradiation : **30 s**
Decay : **300 s**
Count sample: **600 s**
Count flux monitor: **120 s**

Typical capacity:
4 samples /hour/detector

Typical capacity:
28 samples /day/detector

Opportunities through Rapid Turnaround

This approach presents a significant opportunity by providing analytical results within a very short turnaround time, targeting a one-day delivery.

Adopted Scenario:

Day 1 (AM): Sample preparation is performed in the morning.

Day 1 (PM): Irradiation takes place in the afternoon, utilizing a tiered approach based on half-life:

Short Half-Life (SHL): Irradiation for a few seconds, followed by a decay period of few minutes, and a counting time of a few minutes.

Medium Half-Life (MHL): Irradiation for one hour in the morning, followed by a few hours or an overnight decay, and a counting time of a few hours.

Day 2 (AM): Data analysis and reporting are completed in the morning, allowing for a swift delivery of results.

Technical Evaluation:

The technical aspects of transitioning away from a long-half-life (LHL) approach should be thoroughly evaluated.

This should be conducted through a SWOT (Strengths, Weaknesses, Opportunities, and Threats) analysis to assess the implications of this strategic shift.

My talk – just some personal experience:

Short Half-Life (SHL) Protocols
Discussion of the SHL protocols.

The Reactor and Threshold Reactions
How the reactor's characteristics influence the process.
The role and importance of threshold reactions in NAA.

The Rabbit System, Detector, and Timing Effects
The importance of the pneumatic "rabbit" system for rapid sample transfer.
The challenges and requirements for the detector.
An analysis of timing effects and their impact on accuracy.

Certified Reference Materials (CRMs)
The role of CRMs in ensuring accuracy and quality control.
Specific challenges of using CRMs with SHL protocols.

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The SHL-protocols

Categories of radionuclides with SHL

Radionuclides are further sub-categorized as follows, with associated typical experimental conditions (protocols):

Protocol	$t_{1/2}$	t_{irr}	t_d	t_c
Ultra Short half-life radionuclide NAA	< 100 s	10–30 s	1–10 s	10–30 s
Short half-life radionuclide NAA	1–60 min	1–10 min	1 s – 30 min	1–30 min
Medium half-life radionuclide NAA	60 min – 2 d	2–15 min	0.5–4 h	0.5–4 h
Long half-life radionuclide NAA	> 2 d	1–24 h	1–30 d	1 h – several d

If we want to report next day we can

go up to 50 elements – a few we will loose

- **SHL:** Al, Ba, Br, Ca, Cl, Cu, Dy, Ge, I, In, Mo, Mg, Mn, Nb, Nd, Ni, Pd, S, Rb, Rh, Ru, Si, Ti, V, Y, Yb, Zr
- **MHL:** As, Au, Cd, Ce, Er, Ga, Ho, Ir, K, La, Lu, Mo, Na, Pd, Pt, Pr, Re, Sb, Sm, Sn, U, W, Zn
- **LHL:** Ag, Co, Cs, Eu, Fe, Gd, Hg, Os, Sc, Sr, Ta, Tb, Te, Th, Tm would be difficult
  - But, a few of these have metastable we hardly use
  - Many have high Q0
  - Often we simply choose LHL because
    - i. we know them very well after a few weeks of decay e.g. Zn, Sb, Ce, …
    - Ii. we get rid of the Na Compton edge
    - Iii. it offers a very fine QC, because we use the dimension of time

Question we should ask ourselves

- In SHL measurements, the count rates are usually lower. What is the precise effect on the detection limits and counting statistics across different sample matrices?

- How should we treat metastable states that are rarely used? what is their actual relevance and impact?

Question we should ask ourselves

- What is the exact contribution of the Na background, and in cases of high Q_0, how would ENAA improve the situation?

- If we lose access to LHL data, what do we sacrifice in terms of intrinsic quality control in NAA?

- Since most blank values are not well established for SHL, what has been our practical experience with SHL radionuclides when using PE vials?

Counting and Treshold Reactions

The counting and Treshold Reactions

- No need for bigger detectors or well detectors, as most of the time we measure not directly on top of the detector for SHL. A 20% to max. 40% HPGe detecors is perfect

- Best would be a transistor reset preamp, but a resistive feedback preamp works fine as well.

- A specific design of the detector lead shield is needed for the transfer tubes.
- Pb shielding and background are less of an issue compared to LHL.

The counting and Treshold Reactions

- In principle, Loss Free Counting (LFC) modules are needed, as there is decay during counting that requires dead time correction.

- This is standard now in digital DCA (e.g., LYNX module of Mirion).
- Our experience is that it works perfectly up to about 80% dead time (several 100k cps), with peak position shift of < 0.5 keV and FWHM degradation up to 0.5 keV , corresponding to less than 1% uncertainty (using the double source method).

- So, it is perfect for SHL INAA, where we often need up to 50–60% dead time.

But, we suffer more from threshold reactions

- In many research reactors (RR), the SHL irradiations have a lower thermal-to-epithermal and a lower thermal-to fast flux ratio. We can use low-flux RR for SHL!
- Take care: PE vials will thermalize epithermal neutrons, but also epithermalize fast neutrons.
- We must determine the “apparent” g/g of the “interferent” X per µg/g of target X. This can range from a correction factor of about 10 up to 1000 in our case.
- Simulations using known cross sections can easily be made, but in our case they were not that accurate. The best way, and it is very easy, is experimental.

But, we suffer more from threshold reactions

- Go for experimental determination. Just irradiate Al (e.g., Al/Au 99.9% Al, Fe 1% Fe, and hyperpure Si) , and we have most of the threshold reactions covered. These don’t suffer much from self-shielding.

- It depends on the matrix:

- Biological samples will suffer less than, for example, alloys or concrete.

- Here, combining INAA and ENAA would also be beneficial.

Most common threshold interference reactions

Element analyzed	Interfering element	Reaction	$t_{1/2}$	Eγ (keV)
F	**Na**	$^{23}Na(n,\alpha)^{20}F$	11.07 s	1633.6
	Mg	$^{24}Mg(n,p)^{24}Na$	14.96 h	1368.6; 2754
Na	**Al**	$^{27}Al(n,\alpha)^{24}Na$	14.96 h	1368.6; 2754
	Al	$^{27}Al(n,p)^{27}Mg$	9.46 min	843.8; 1014.4
Mg	**Si**	$^{30}Si(n,\alpha)^{27}Mg$	9.46 min	843.8; 1014.4
	Si	$^{28}Si(n,p)^{28}Al$	2.25 min	1779.0
Al	**P**	$^{31}P(n,\alpha)^{28}Al$	2.25 min	1779.0
Ti	**V**	$^{51}V(n,p)^{51}Ti$	5.76 min	320.1
	Cr	$^{52}Cr(n,p)^{52}V$	3.74 min	1434.1
V	**Mn**	$^{55}Mn(n,\alpha)^{52}V$	3.74 min	1434.1
Mn	**Fe**	$^{56}Fe(n,p)^{56}Mn$	2.579 h	846.8; 1810.7; 2113.1
	Co	$^{59}Co(n,\alpha)^{56}Mn$	2.579 h	846.8; 1810.7; 2113.1
		$^{66}Zn(n,p)^{66}Cu$	5.12 min	1039.2
Cu	**Zn**	$^{64}Zn(n,p)^{64}Cu$	12.7 h	1345.8
	Cu	$^{65}Cu(n,p)^{65}Ni$	2.52 h	1481.8
Ni	**Zn**	$^{68}Zn(n,\alpha)^{65}Ni$	2.52 h	1481.8
Rb	**Sr**	$^{88}Sr(n,p)^{88}Rb$	17.77 min	898; 1836

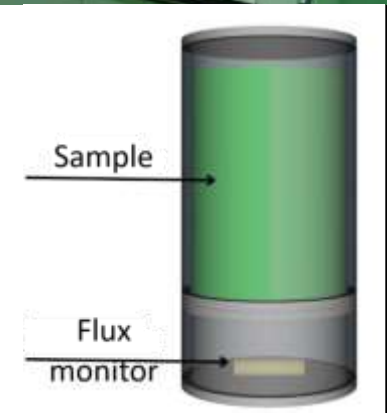
The Rabbit - Timing effects

Our Rabbit

- We work with pneumatic PTS system, partially automated– aluminum tubing

- Samples are sealed in PE capsules (about 10 mm in diameter and 22 mm in length)

  - We go for immediate counting on the HPGe for Al and V, but there is Na in the capsules leading to threshold interference corrections.



Our Rabbit

- We go to a Pb-window shielded fumehood for all other radionuclides where we manually dismantle

- The travel time going in and out to the reactor core is about 5s.

- Other issue is that there is flux gradients from axial and radial, leading to the fact that we have to sandwich every sample with fluxmonitors, what we also did for LHL

Timing effects

- Synchronisation between rabbit PTS and Couting PC can be a problem.
- Travel time going in/out of the core and flux variation during travelling has impact on saturation
- Travel time going out of the core has impact on the decay
- Can be minimised using standards
- Best determined experimentally by using different irradiation and decay times and simulating travel in and travel out
  - Travel time to the detector is about a 5 min (depends on the induced activity of the sample)

04

CRM’s

CRM’s

- To make a literature survey which CRM’s would be beneficial for different matrix groups biological, geological, constrution, …

SMELS Type I & II

- Big difference in Q_0 – so allows for QC of reactor channel calibration
- Big difference in E_γ – so allows for QC of detector calibration
- Big difference in $T_{1/2}$ – so allows for QC of timing

SMELS 1			
Element	**X (n,x) Y**	**$T_{1/2}$**	**Eγ (keV)**
Au	^{197}Au (n, γ) ^{198}Au	2.695 d	411.8
Cl	^{37}Cl (n, γ) ^{38}Cl	37.24 min	1642.7, 2167.4
Cs	^{133}Cs (n, γ) ^{134m}Cs	**2.903 h**	**127.5**
Cu	^{65}Cu (n, γ) ^{66}Cu	5.12 min	1039.2
I	^{127}I (n, γ) ^{128}I	24.99 min	442.9
La	^{139}La (n, γ) ^{140}La	1.678 d	328.8, 487.0, 1596.2
Mn	^{55}Mn (n, γ) ^{56}Mn	2.579 h	846.8, **1810.7**
V	^{51}V (n, γ) ^{52}V	**3.75 min**	1434.1

SMELS 2			
Element	**X (n,x) Y**	**$T_{1/2}$**	**Eγ (keV)**
As	^{75}As (n, γ) ^{76}As	26.24 h	559.1
Au	^{197}Au(n, γ) ^{198}Au	2.695 d	411.8
Br	^{81}Br (n, γ) ^{82}Br	35.30 h	554.3, 619.1, 776.5
Ce	^{142}Ce (n, γ) ^{143}Ce	33.10 h	293.3
Mo	^{98}Mo (n, γ) ^{99}Mo	65.94 h	181.1, 739.5.
Mo	^{98}Mo (n, γ) ^{99m}Tc	**6.01 h**	**140.47**
Pr	^{141}Pr (n, γ) ^{142}Pr	19.12 h	**1575.6**
Sb	^{121}Sb (n, γ) ^{122}Sb	2.724 d	564.2
Yb	^{174}Yb (n, γ) ^{175}Yb	**4.185 d**	282.5, 396.3
Zn	^{68}Zn (n, γ) ^{69m}Zn	13.76 h	438.6

- **Conclusion**

- Six decades of experience in NAA has resulted in commonly applied analytical protocols, with measurements at about two to seven days and at three weeks after neutron irradiation. NAA with short half-life radionuclides enables an analysis turnaround time of one working day in principle.
- It also enables the detection of many important elements that cannot otherwise be measured with NAA based on longer half-lives. It may therefore provide competitive and, in some cases, even unique opportunities for an NAA facility. This opportunity is not fully seized by NAA laboratories due to the technical and analytical difficulties involved.
- This publication addresses all aspects of the implementation and optimization of NAA with short half-life radionuclides.

Royaume du Maroc

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Thank you for your attention

Merci pour votre aimable attention

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