First X-ray fluorescence spectra with LEAPS-INNOV HPGe detector using synchrotron radiation

N. Goyal (SOLEIL Synchrotron) and A. Balerna (INFN DAΦNE-Light)

on behalf of the LEAPS - INNOV EU project - WP2 XAFS-DET collaboration

High Precision X-ray Measurements 2025 - INFN LNF - 17 June 2025

LEAPS

LEAPS – the **League of European Accelerator based-Photon Sources** unites all **Synchrotron** and **Free Electron Laser** accelerators in Europe.

It was created in **2017** with the primary goal of actively and constructively promote and ensure the **quality** and **impact** of **fundamental, applied** and **industrial research** carried out at its members.

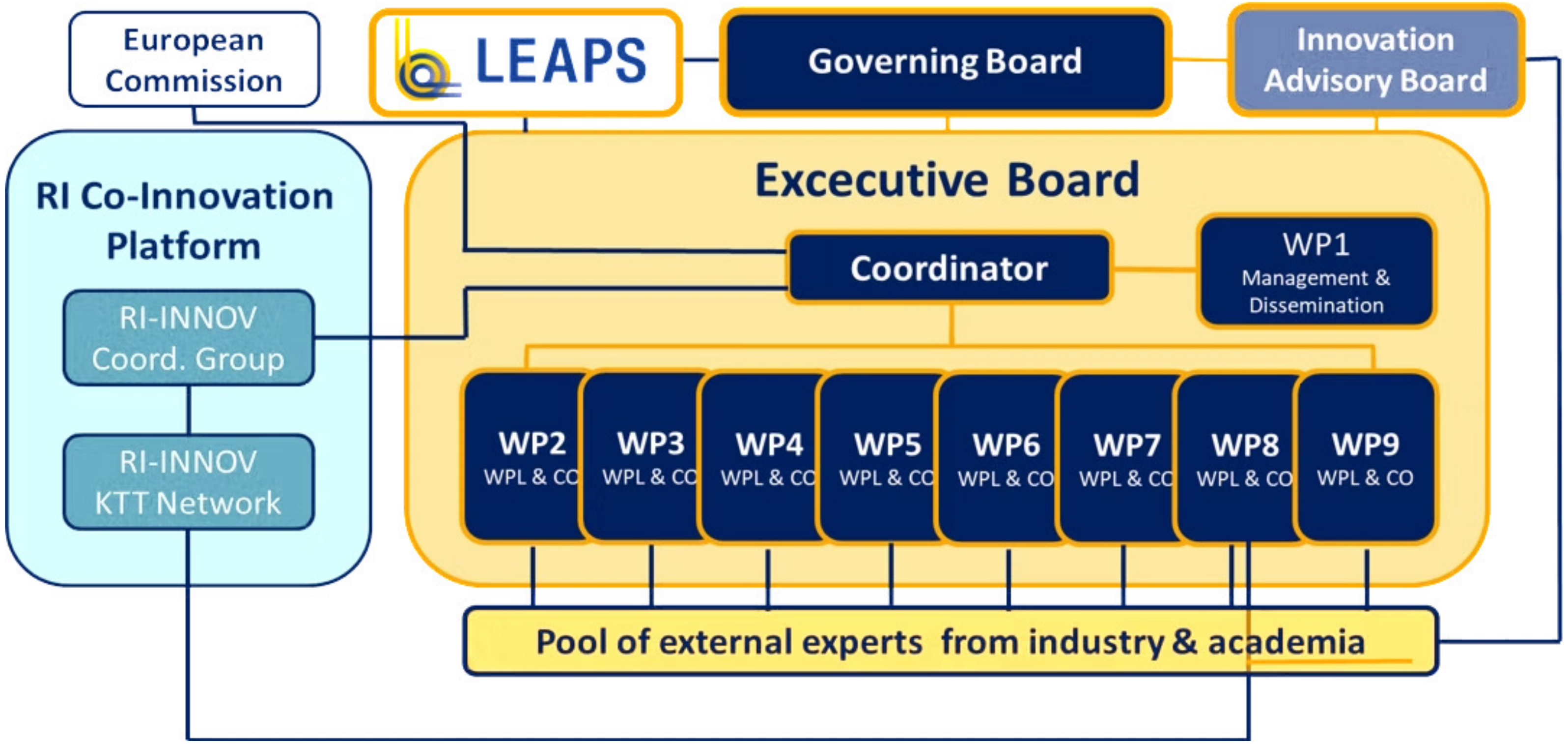
Science at Synchrotron Radiation and Free Electron Laser facilities play an essential role in the discovery and characterisation of advanced materials, biomaterials and living matter and Europe has achieved global leadership in this field.

LEAPS research infrastructures serve a **very broad scientific community of more than 35,000 researchers in Europe** and attract some of the brightest minds worldwide.

SESAME · LENS · Canadian Light Source / Centre canadien de rayonnement synchrotron · ESUO EUROPEAN SYNCHROTRON AND FEL USER ORGANISATION

https://www.leaps-initiative.eu/

LEAPS-INNOV



https://www.leaps-innov.eu/

The **LEAPS-INNOV pilot project** focusses on the **implementation of new strategies** and **activities for long-term partnerships between European industry and the European light sources, synchrotrons and free-electron lasers,** with their tens of thousands of users.

LEAPS-INNOV aims at **kick-starting the implementation of the LEAPS Technology Roadmap**. It will offer joint **technological developments and advanced research capabilities with LEAPS members for industry as collaborator, supplier and user.**

April 2021 - November 2025

WP2 XAFS-DET (LEAPS-INNOV)

Development of High Throughput X-ray Spectroscopy Detector System

Work Package Leaders: E. Gimenez-Navarro (DIAMOND), F.-J. Iguaz- Gutierrez (SOLEIL)

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Facilities involved: DIAMOND[f], SOLEIL[a], DESY[h], ALBA-CELLS[e], ELETTRA[k], ESRF[g], INFN DAΦNE-Light[c], PSI[m], SOLARIS[i], MAX IV[d], XFEL[b]

The aim of this work package is to push germanium detectors performance beyond the state-of-the-art, and for instance, **enhance the throughput per unit area for X-ray spectroscopy by developing new multi-element monolithic detectors with reduced element size.**

Shrinking the element size entails challenges such as the development of miniaturized front-end electronics and the use of advanced pulse processors to avoid collimators in front of the elements.

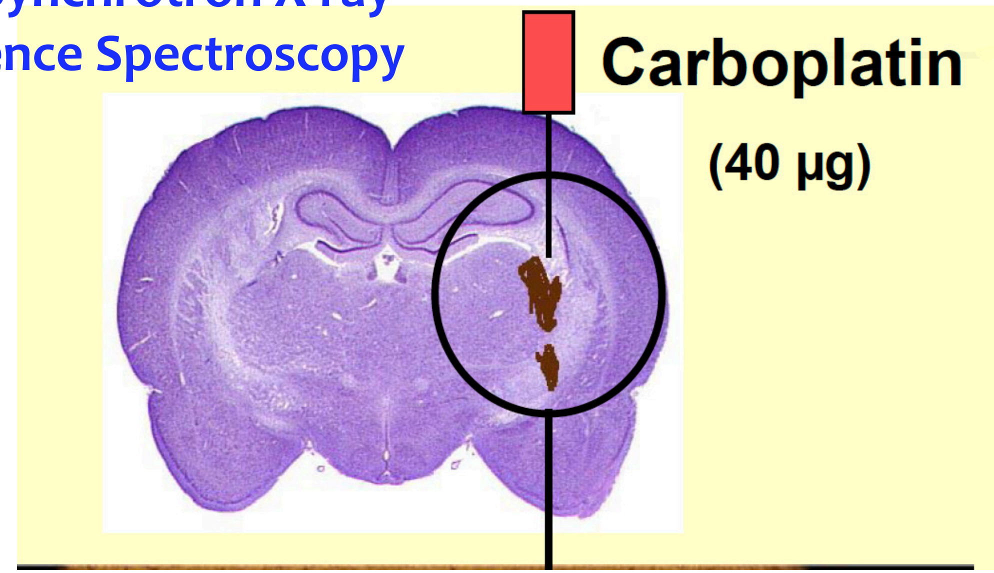
The work package will develop a prototype with a limited number of channels, but the **technology developed within this project will enable to scale up the channels in future developments.**

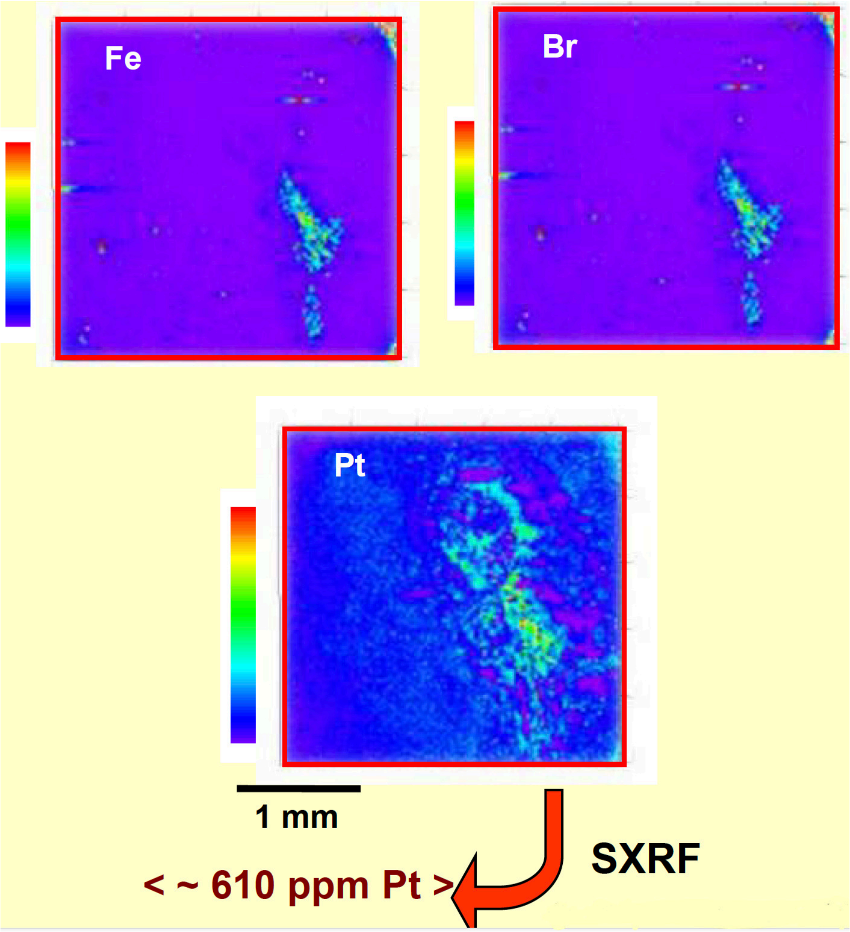
Applications

XRF and XAFS

Cancer Research - Metal detection in cells and tissues -carboplatin detections in brain tumor

SXRF – Synchrotron X-ray Fuorescence Spectroscopy

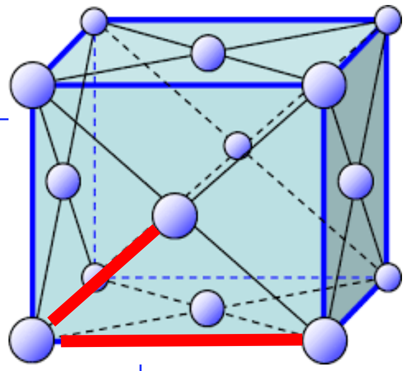




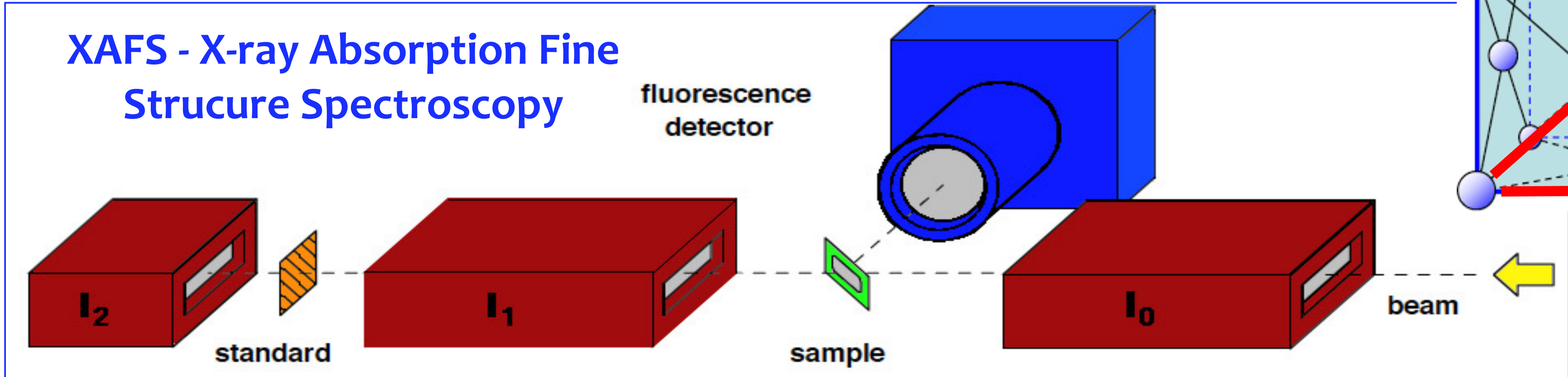
Cancer Research - Metal detection in cells and tissues – carboplatin detections in brain tumor –synchrotron photoactivation - 78.4 keV Pt K-edge

S. Bohic (INSERM 836 Team 6 - Grenoble Institute of Neuroscience) - Applications to metal detection in biological samples - School on X-ray imaging techniques - ESRF 2007

XRF and XAFS

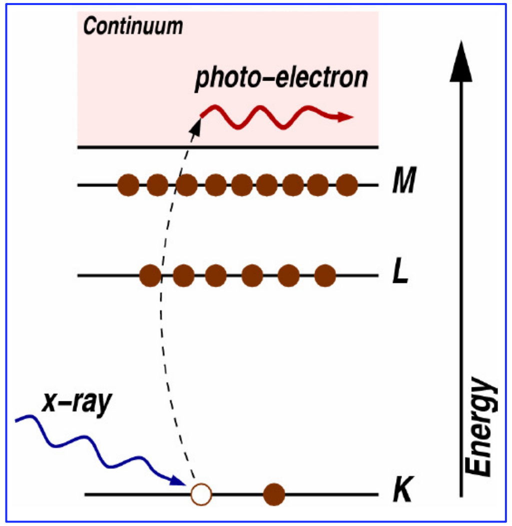


XAFS - X-ray Absorption Fine Strucure Spectroscopy

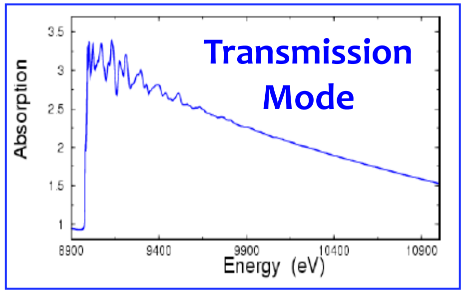


XAFS can be measured either in Transmission or Fluorescence mode.

X-ray absorption

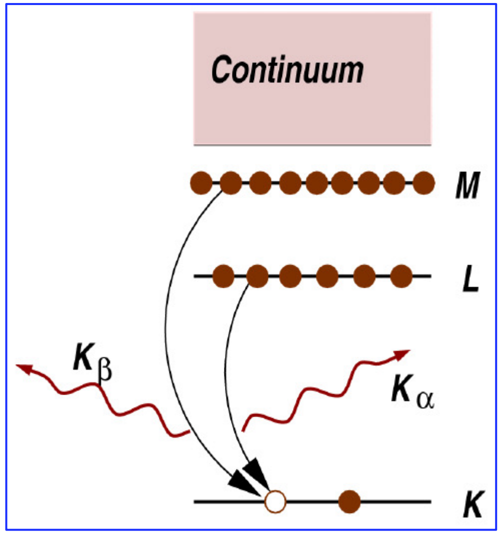


$$\mu(E)x = \ln\left(\frac{I_0}{I_1}\right)$$

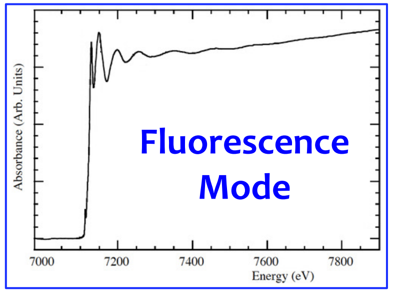


The probability of fluorescence emission is directly proportional to the absorption probability.

X-ray fluorescence

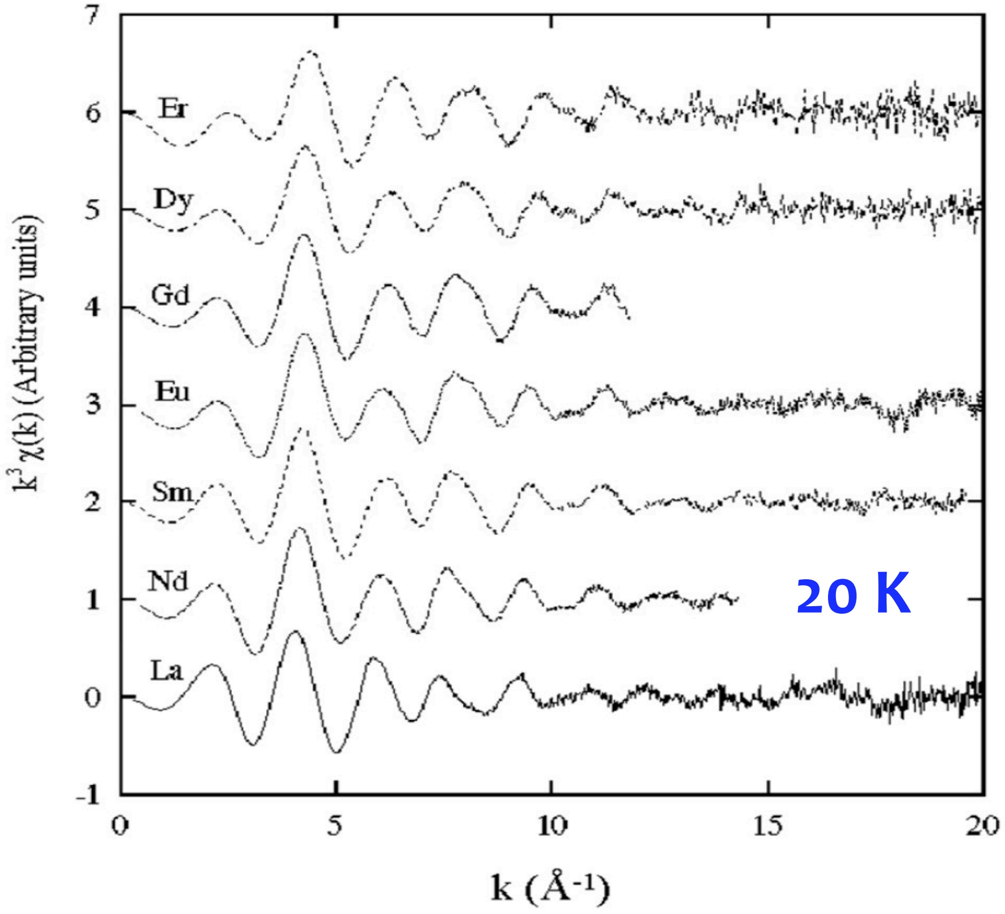


$$\mu(E)x = \left(\frac{I_F}{I_0}\right)$$



A rare-earth K-edge EXAFS study of rare-earth phosphate glasses, $(R_2O_3)x(P_2O_5)_{1-x}$, x = 0.187-0.239, R = La, Nd, Sm, Eu, Gd, Dy, Er.

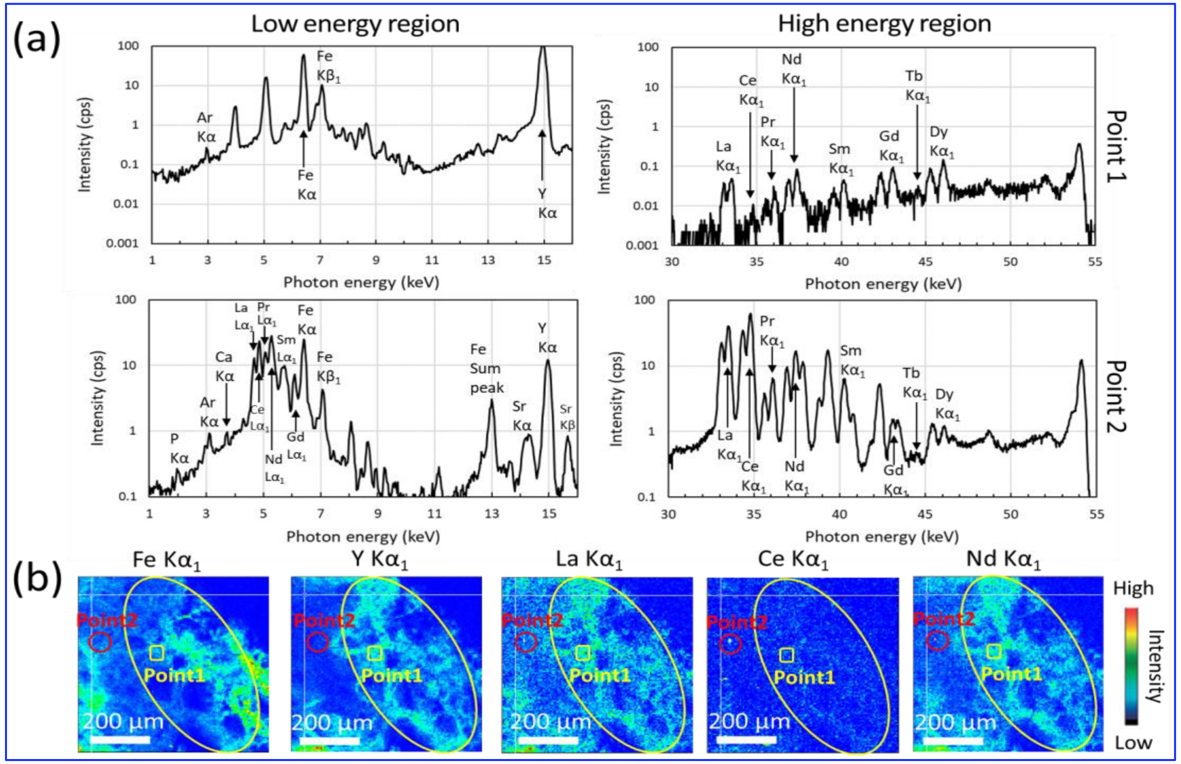
K-edge EXAFS study of rare-earth phosphate glasses results are compared to those obtained from an analogous rare-earth **L_{III}-edge EXAFS** (5.483-8.358 keV) **study**. Results show that the **use of the much higher energies of the rare-earth K-edge (38.925 - 57.486 keV)** **avoids** the **double-electron excitation problems that are associated with the rare-earth L_{III}-edge** **EXAFS in the dynamic range of interest.** EXAFS fitting and deconvolution simulations show that the **large core hole lifetimes associated with the rare-earth K-edge do not significantly detract from the results.**



Jacqueline M Cole *et al* 2001 *J. Phys.: Condens. Matter* **13** 6659

Development of High-Energy μ-X-ray Fluorescence and X-ray Absorption Fine Structure for the Distribution and Speciation of Rare Earth Elements in Natural Samples

Micro-X-ray fluorescence and X-ray absorption fine structure (μ-XRF-XAFS) is one of the **most powerful tools to identify the distribution and speciation of trace elements in natural samples** with μm spatial resolution. However, **conventional μ-XRF-XAFS studies** applied to **rare earth elements** (REEs: lanthanide elements + Y in this study) are mainly **limited to their L-edges and L lines (except for Y)** that are subject to **strong interferences from other elements (mainly transition metals).** μ-XRF-XAFS was extended to the higher energy region (**HE-μ-XRF-XAFS**) by using an incident X-ray microbeam (size: ca. **1 × 1 μm²**) between **38 and 54 keV to realize K-edge excitation lanthanide** analysis without interferences from other elements at the **BL37XU beamline, SPring-8 (Japan).** This method enables us to **simultaneously analyse** (i) **REE patterns (from La to Dy)**, (ii) **XAFS spectra**, and (iii) **μm-scale distribution of each REE in the natural sample**.



M. Nagasawa et al. *Minerals* **2023**, *13*(6), 746

Why XAFS-DET?

To push HPGe detectors' performance beyond the state-of-the-art.

XAFS-DET

The **high brightness** resulting from **recent upgraded synchrotron radiation facilities** opens the way for a large range of experiments, where **detectors play a key role in the techniques and methods developed to fully exploit the upgraded synchrotrons.**

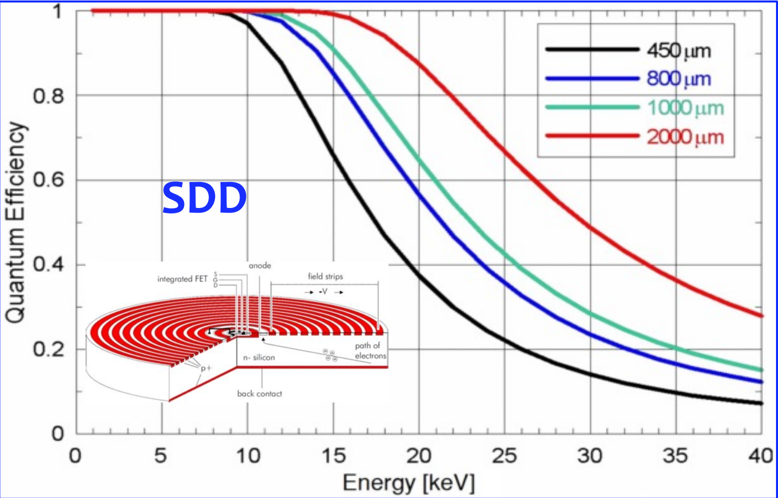
To **measure more challenging samples** and to **cope with the very high photon flux of the current** and **future** (**diffraction limited**) **sources**, technological developments of detectors are necessary.

These new **germanium detectors** aim at improving several technological aspects to **detect efficiently photons** of considerable **higher energy with respect to silicon detectors**.

The objective of this project consists in **pushing the detector performance beyond the state-of-the-art**.

For **low energies (<20keV), Silicon is a near-ideal detector material** offering advanced processing technologies including the fabrication of on-detector low-noise electronics. **Higher energies require Germanium**, but detectors made from this material are **unlikely ever to reach the sophistication of silicon devices due to the lack of a large-scale market to spur the needed developments**.

J Morse, Detector Unit - ESRF 2010. Energy resolving semiconductor detectors for X-ray spectroscopy



Ge SSD detectors

Fano energy resolution, leakage current (noise) $\frac{\Delta E}{E} \propto \frac{1}{\sqrt{N}}$

Signal development time (max. counting rate)

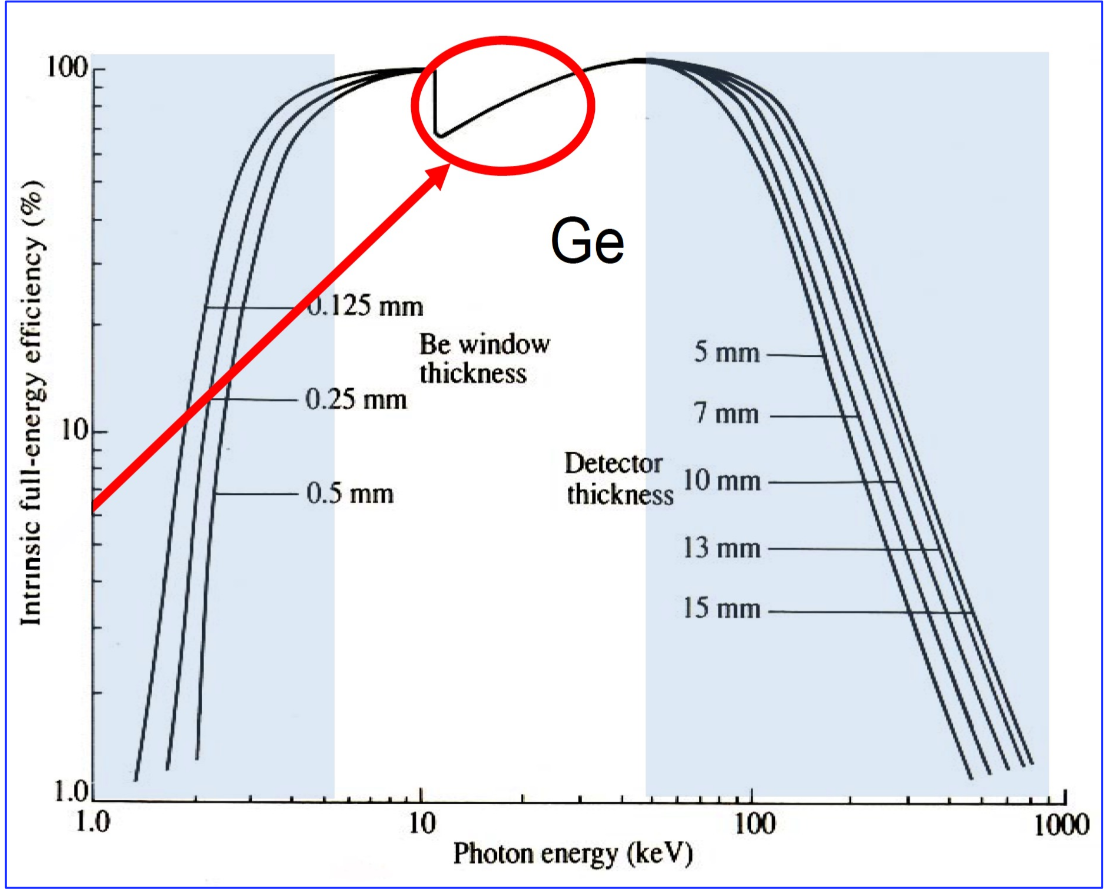
stopping power, X-ray absorption length

Mono-elemental crystals-excellent charge transport

Binary and ternary compounds - trapping of charge during drift (signal loss)

Material	Z	Bandgap [eV]	Mobility [cm^2/Vs] electrons	Mobility [cm^2/Vs] holes	Density g/cm^3
Si	14	1.1	1350	480	2.3
Ge	32	0.7	3800	1800	5.3
Diamond	6	5.5	4500	3500	3.5
GaAs	31-33	1.5	8600	400	5.4
AlSb	13-51	1.6	200	700	4.3
GaSe	31-34	2.0	60	250	4.6
CdSe	48-34	1.7	50	50	
CdS	48-16	2.4	300	15	4.8
InP	49-15	1.4	4800	150	
ZnTe	30-52	2.3	350	110	
WSe_2	74-34	1.4	100	80	
BiI_3	83-53	1.7	680	20	
Bi_2S_3	83-16	1.3	1100	200	6.7
Cs_3Sb	55-51	1.6	500	10	
PbI_2	82-53	2.6	8	2	6.2
HgI_2	89-53	2.1	100	4	6.3
CdTe	48-52	1.5	1100	100	6.1
CdZnTe	48-30-52	1.5-2.4			

J. Morse ESRF - 1st EIROforum School on Instrumentation, Cern 11-15 May 2009



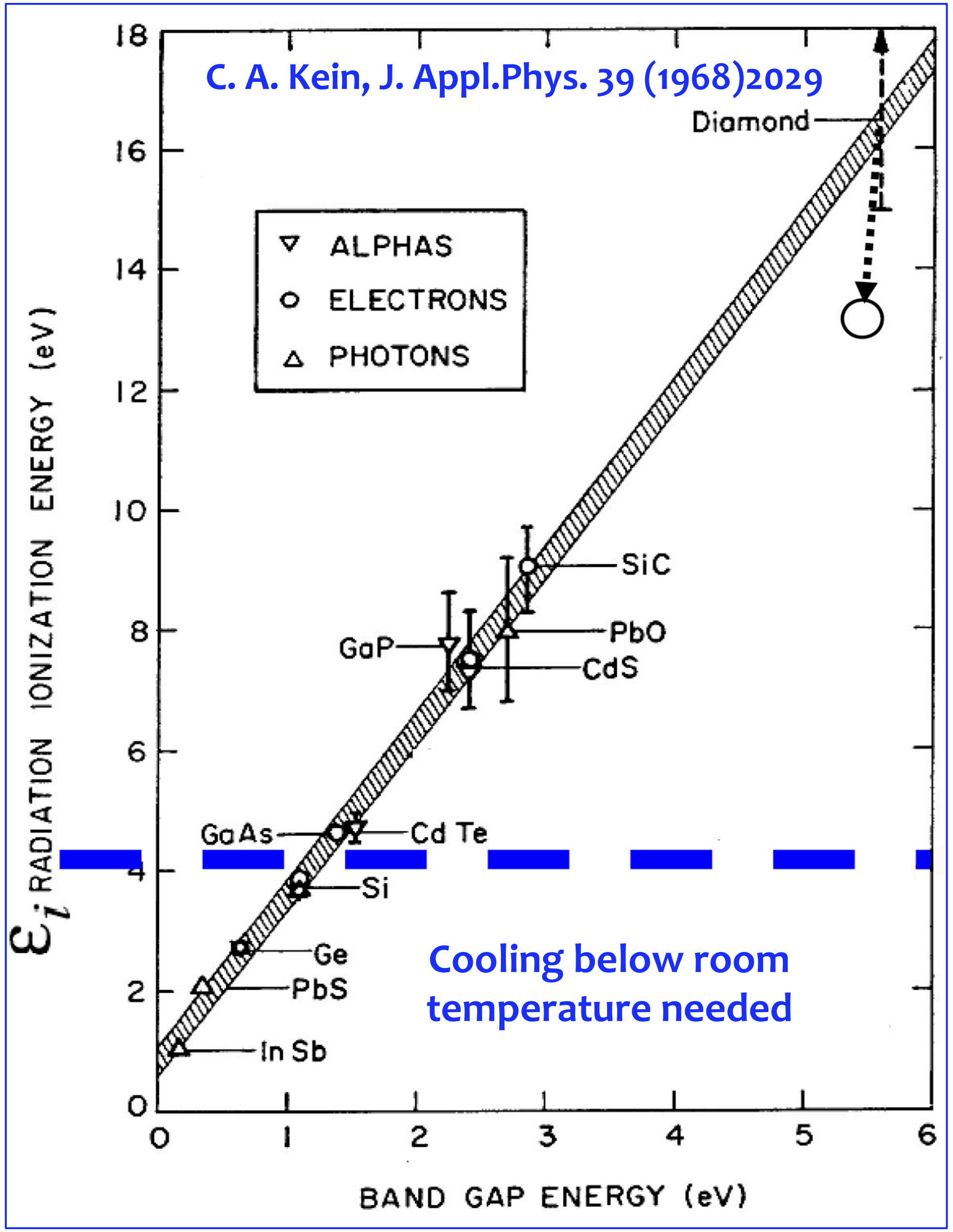
New **germanium detectors** aim at improving several technological aspects to **detect efficiently photons** of considerable **higher energy with respect to silicon detectors**.

Ge SSD detectors

Because **germanium has relatively low band gap**, these detectors must be **cooled** to **reduce the thermal generation** of charge carriers (thus **reverse leakage current**). **Leakage current induced noise destroys the energy resolution of the detector**. **Liquid nitrogen, which has a temperature of 77 K is the common cooling medium for such detectors**.

↓

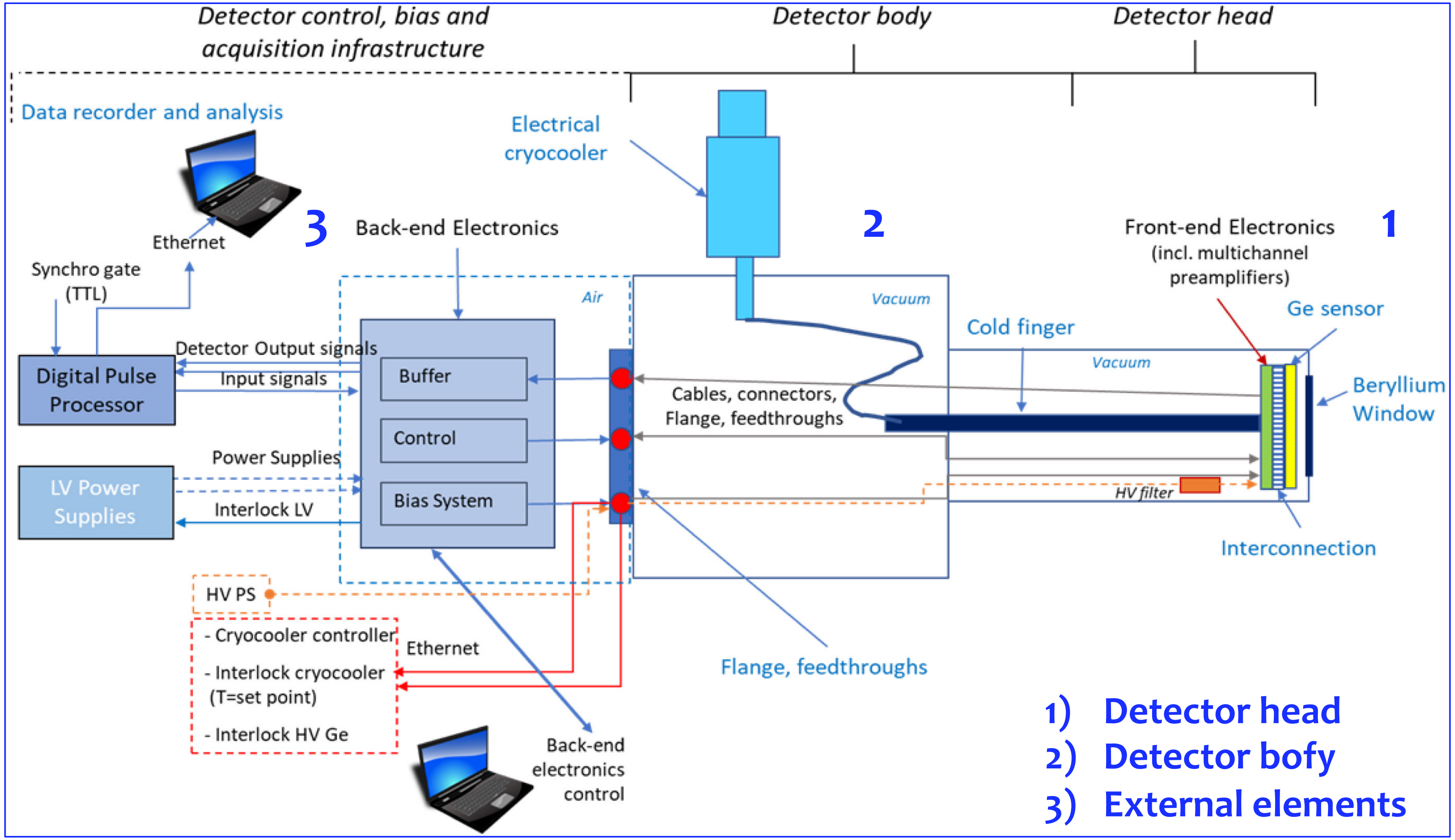
Moving to **electrical cryocooling**



WP2 XAFS-DET
2021-2025

From design and simulation phase to assembled stage and tests

XAFS-DET: A new high throughout X-ray spectroscopy detector system developed for synchrotron applications

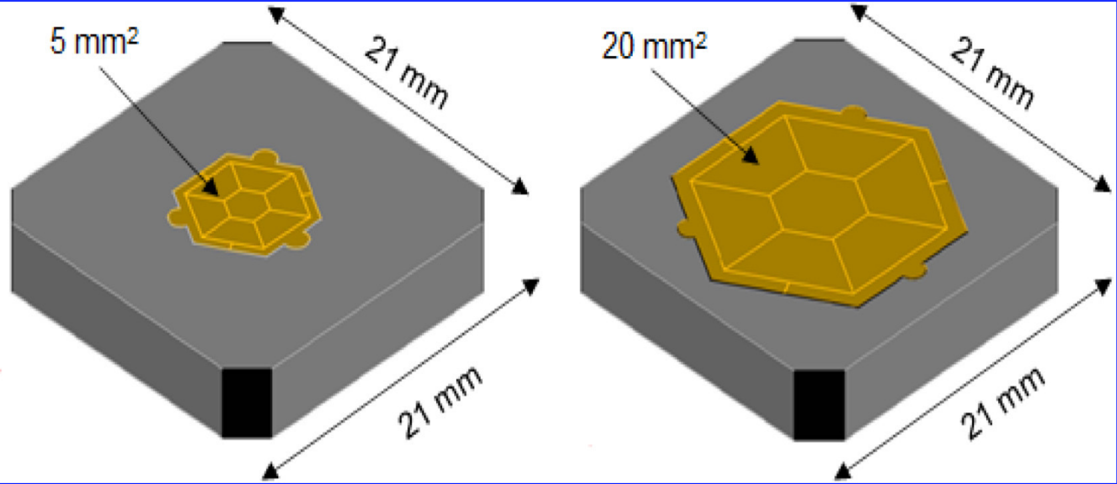


1) Detector head
2) Detector bofy
3) External elements

2023

F. Orsini et al. NIMA 1045 (2023) 167600

XAFS-DET



Mirion has the capability to **make segmented planar Ge detectors** using **advanced photolithographic techniques:** **many pixel detectors are formed in a single slice of germanium.**
The **packing density** defined as the **active detector area divided by the total area** in monolithic detectors, which have no dead space between elements, is **virtually 100%.** The packing density of discrete array detectors ranges from about 35 to 55%. **Packing density is an important factor in applications requiring an optimized solid angle and best fit to detection area.**

https://www.mirion.com/

Table 1. Main requirements for the XFAS-DET detector prototype.

X-ray energy range	From 5 to 100 keV
Number of pixels	10
Pixel size and thickness	Small pixel version of 5 mm^2 and large pixel version of 20 mm^2, both versions are 4mm thick
Energy resolution	<200 eV FWHM at 5.9 keV and an Input Count Rate of 100 kcps/pixel and a shaping time (or equivalent) of 1 μs
Detector efficiency	>90% in the required energy range
Input Count Rate (ICR) range per unit area	From 20 $kcps/mm^2$ up to 250 $kcps/mm^2$
Dead time (%) at 5.9 keV	30% at 1 Mcps and maintained at reasonable level at high energy (20–100 keV)
Peak to Background Ratio	>1000 at 5.9 keV, defined as the ratio between the counts in the peak channel and the average background
Detector temperature	77 K (detector head and body)
Detector vacuum	1×10^{-7} mbar
Input Window	Beryllium window
Germanium sensor collimator	Multi-layer collimator **- Mo collimator - 3mm**

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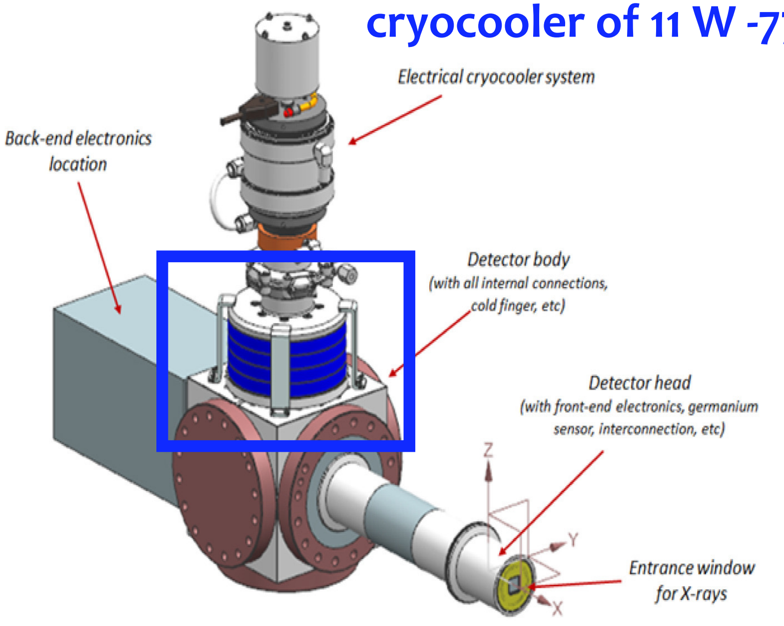
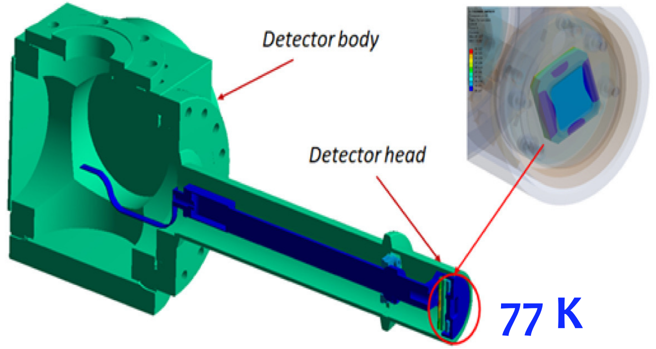
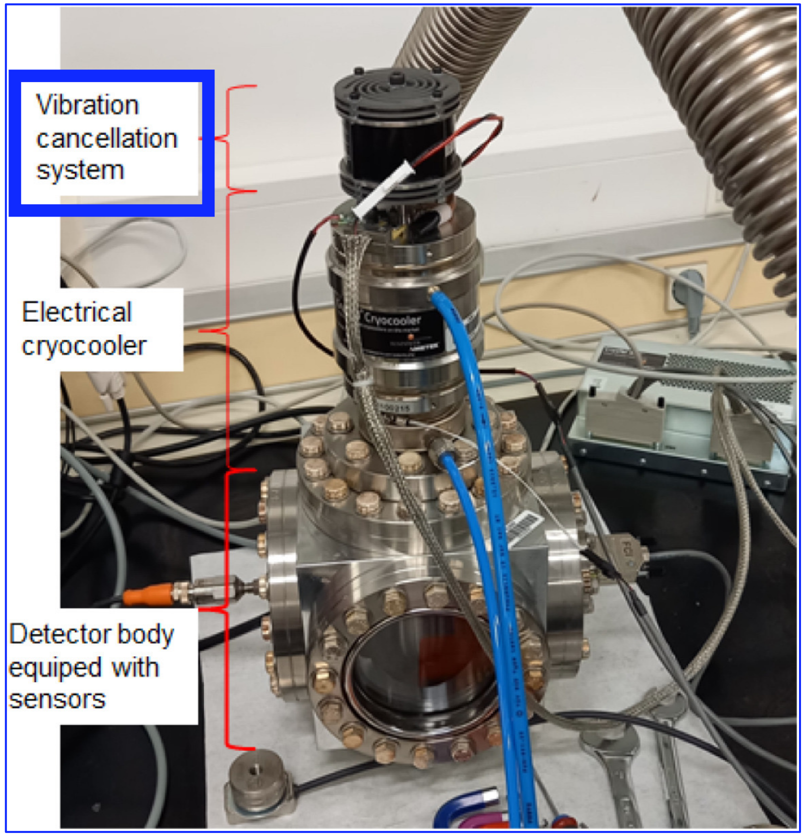
XAFS-DET

SunPower electrical cryocooler of 11 W -77K





M. Quispe - ALBA



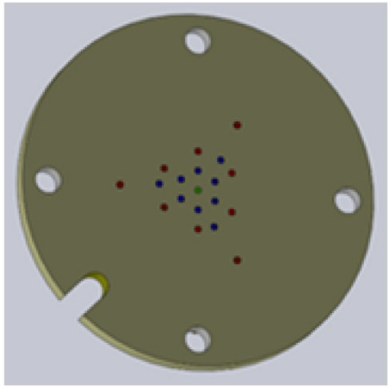
Thermo-mechanical simulations – for a detector that should work at **77 K using a cryocooler** being sure that **no mechanical vibrations are introduced by the electrical cryocooling**.

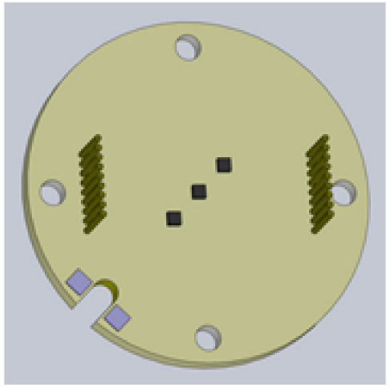
https://www.sunpowerinc.com/

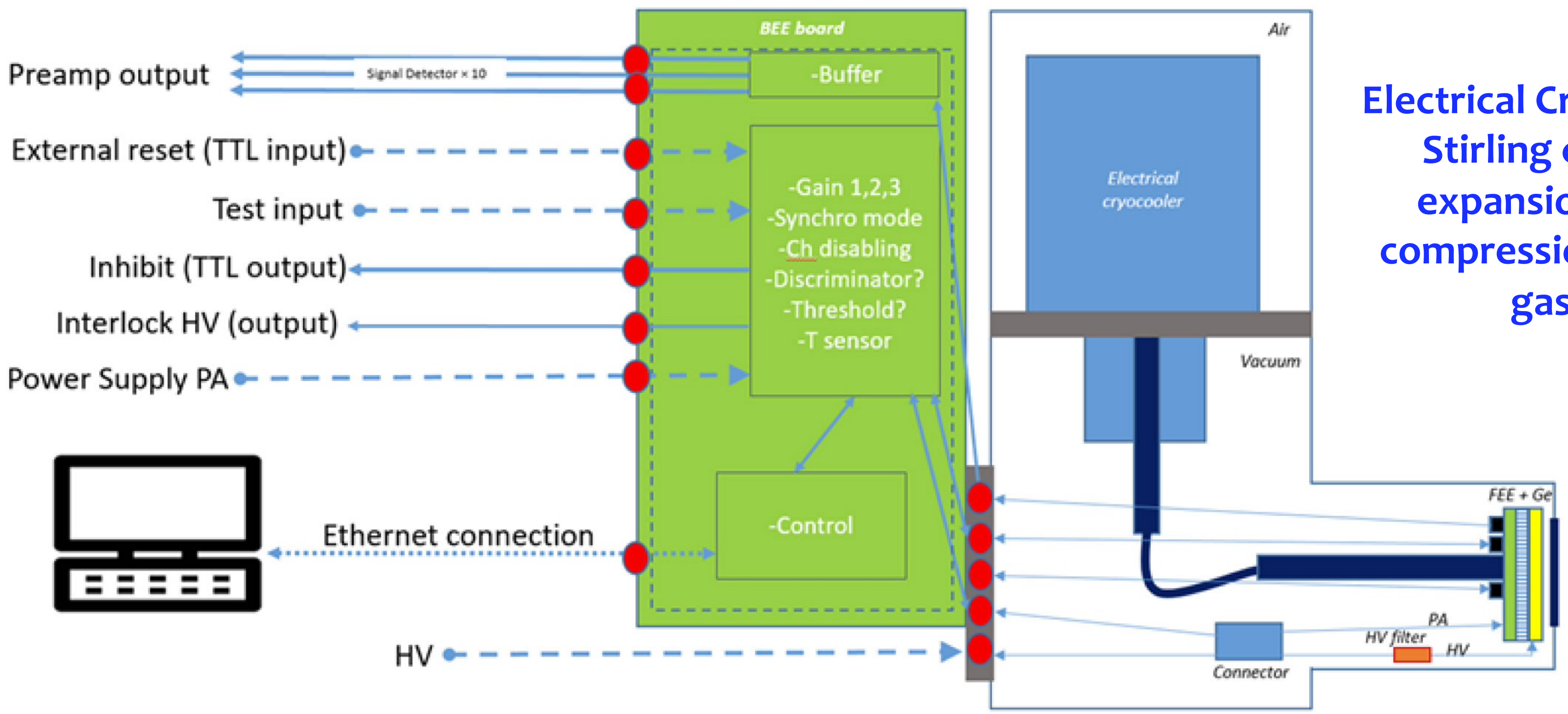
F. Orsini et al. NIMA 1045 (2023) 167600

XAFS-DET

2023







Electrical Cryocooler Stirling cycle - expansion and compression of He gas.

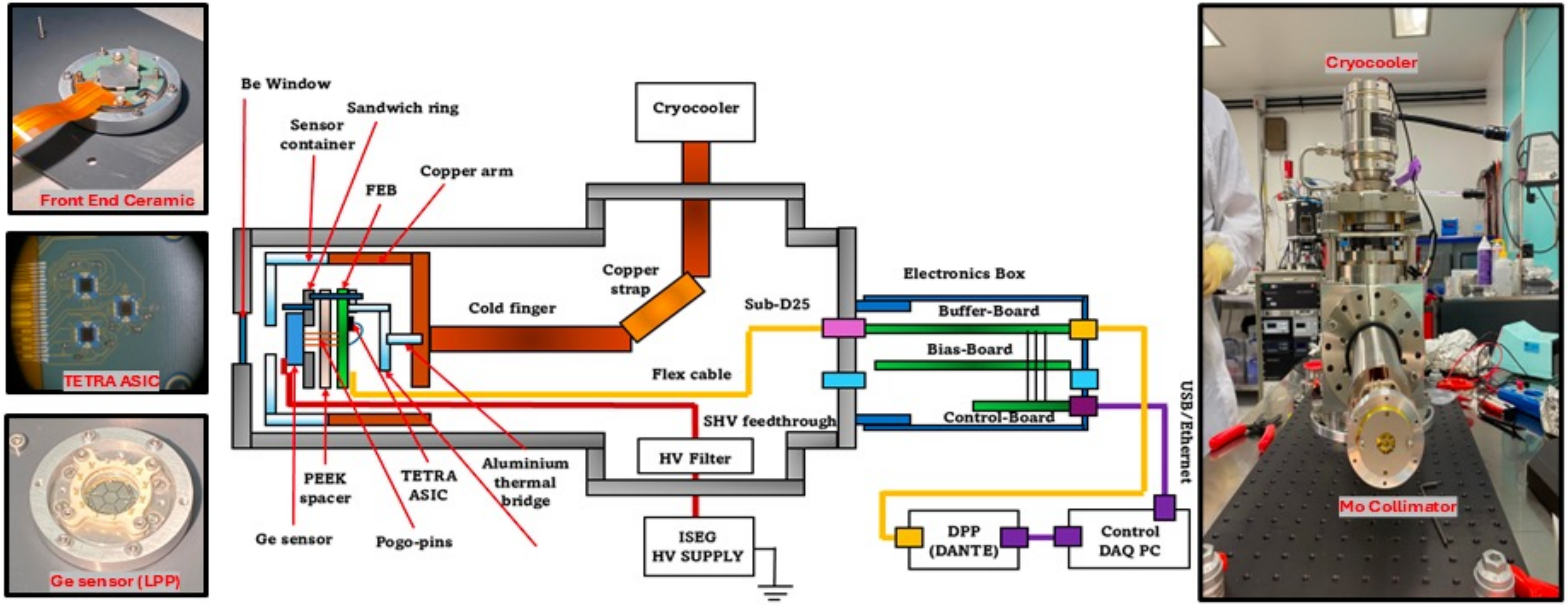
Overview of the front-end board electronic (left): top side of the board matching both the small and large pixels arrangement (top-left) and its back side (bottom-left); **Schematic overview of the functionalities of the back-end electronics** (right).

https://www.xglab.it/

F. Orsini et al. NIMA 1045 (2023) 167600

XAFS-DET electronics chain is composed of three parts: a **multi-channel integrated preamplifier** (TETRA ASIC), a **front-end board** (FEC) and a **back-end board** (BEC). The full chain has been fabricated by **XGLab - Bruker Nano Analytics Division.**

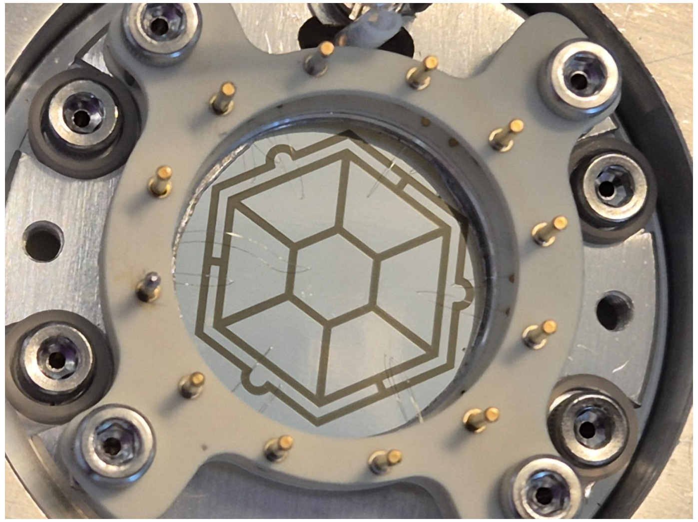
XAFS-DET 2024



Schematic overview of the XAFS-DET detector setup, illustrating the **Ge sensor (LPP-Large Pixel Pitch) integration with the front-end and back-end electronics**, **cryocooler**, and **data acquisition (DAQ) architecture**. The **Ge sensor is connected to the TETRA ASIC-based Front-End ceramics** (**FEC**), which **interfaces with the Back-End Board** (**BEB**) for signal amplification and buffering. The **DANTE Digital Pulse Processor** (DPP) was used **instead of Xspress4 for the first integration tests** to evaluate electronic noise and detector response. The **setup was operated at a temperature of 77 K.**

N. Goyal et al. *Next Generation Multi-element monolithic Germanium detectors for Spectroscopy: First integration at ESRF facility* (**SRI 2024**)

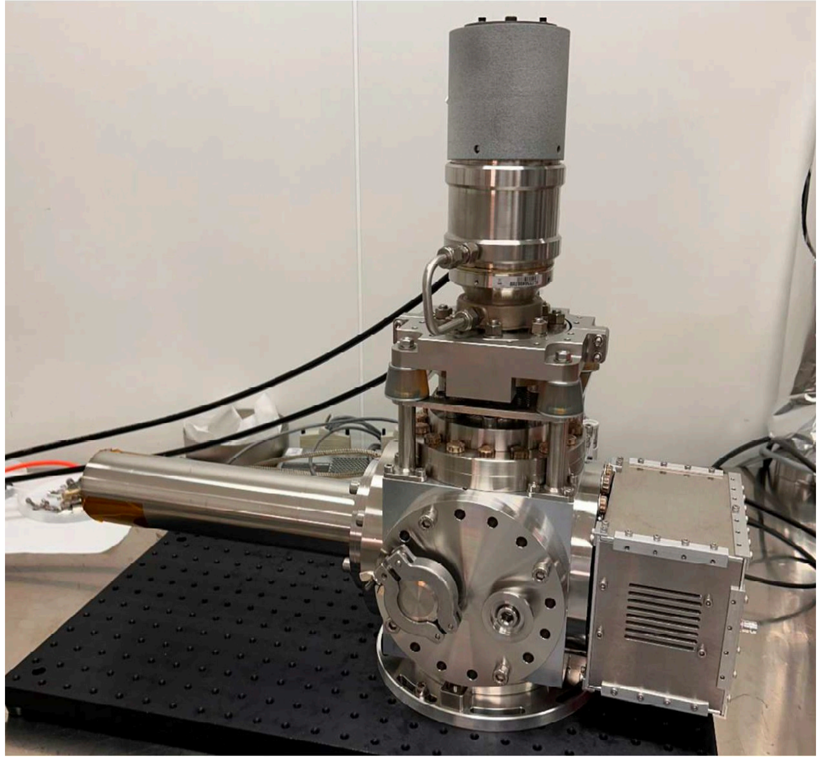
XAFS-DET



LPP HPGe crystal mounted.
R. Alberts - DIAMOND

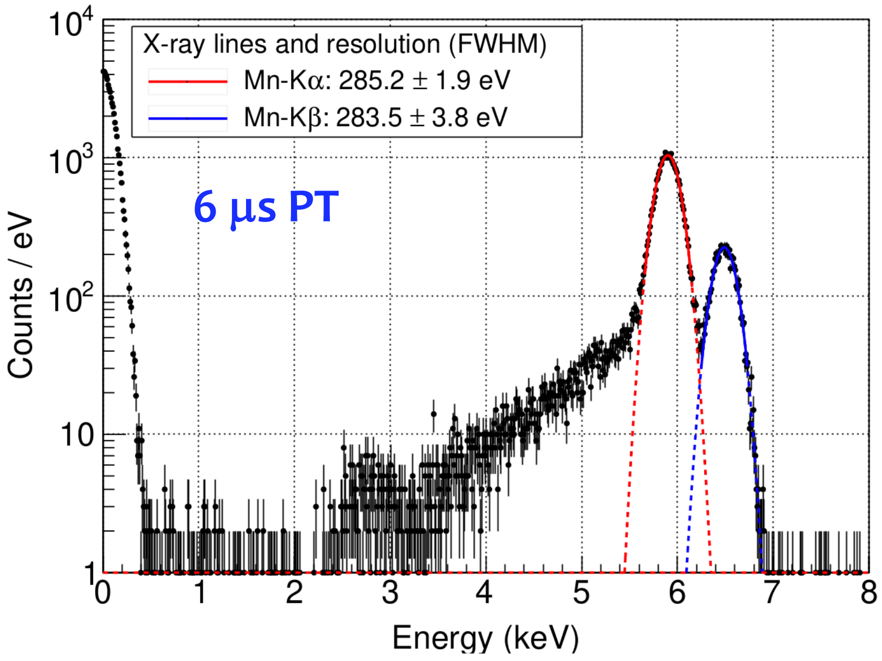
LPP 4.1 mm thick
SPP 4.4 mm thick

47kg!



Assembled HPGe detector
C. Cohen - ESRF

Tests initially performed using radioactive sources ^{55}Fe, ^{241}Am and conventional X-ray tubes.



Measured energy spectrum of ^{55}Fe radioactive source.
N. Goyal -SOLEIL

Goal: 180 eV at 5.9 keV – ICR 1Mcps – 1 μs peaking time

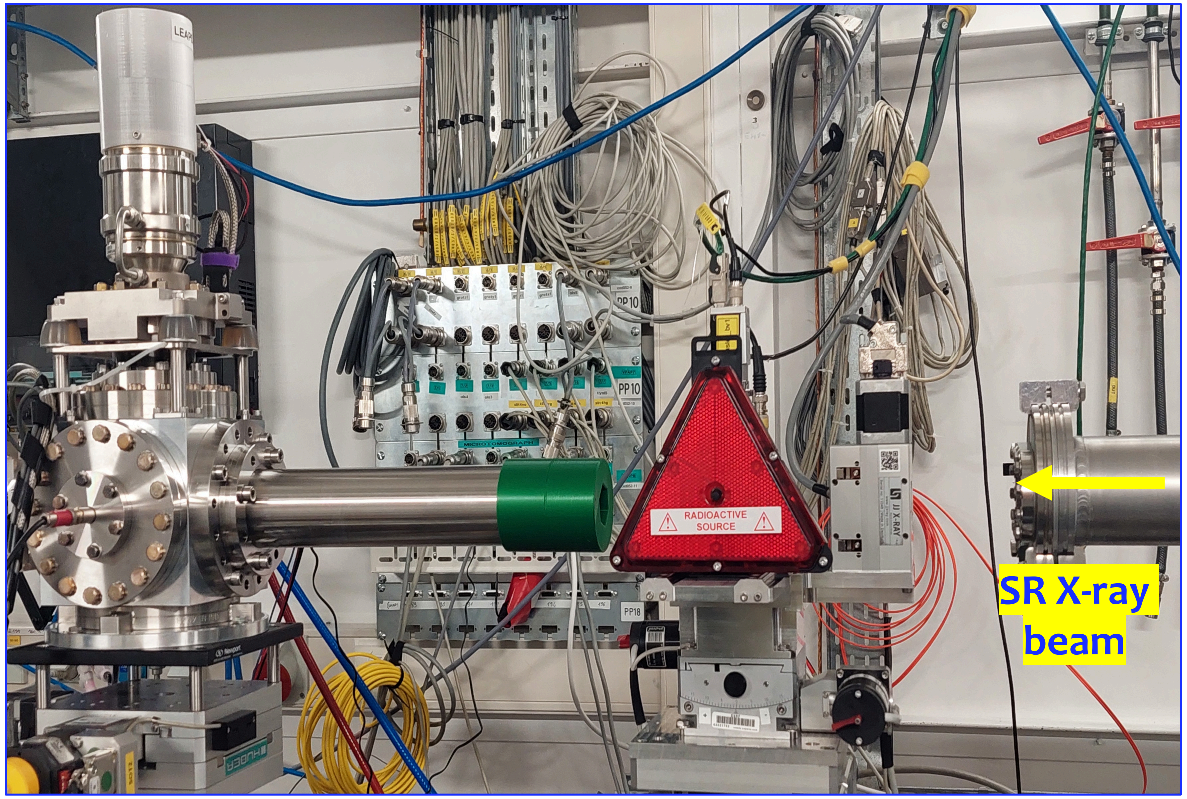
First X-ray fluorescence spectra with LEAPS-INNOV HPGe detector using synchrotron radiation

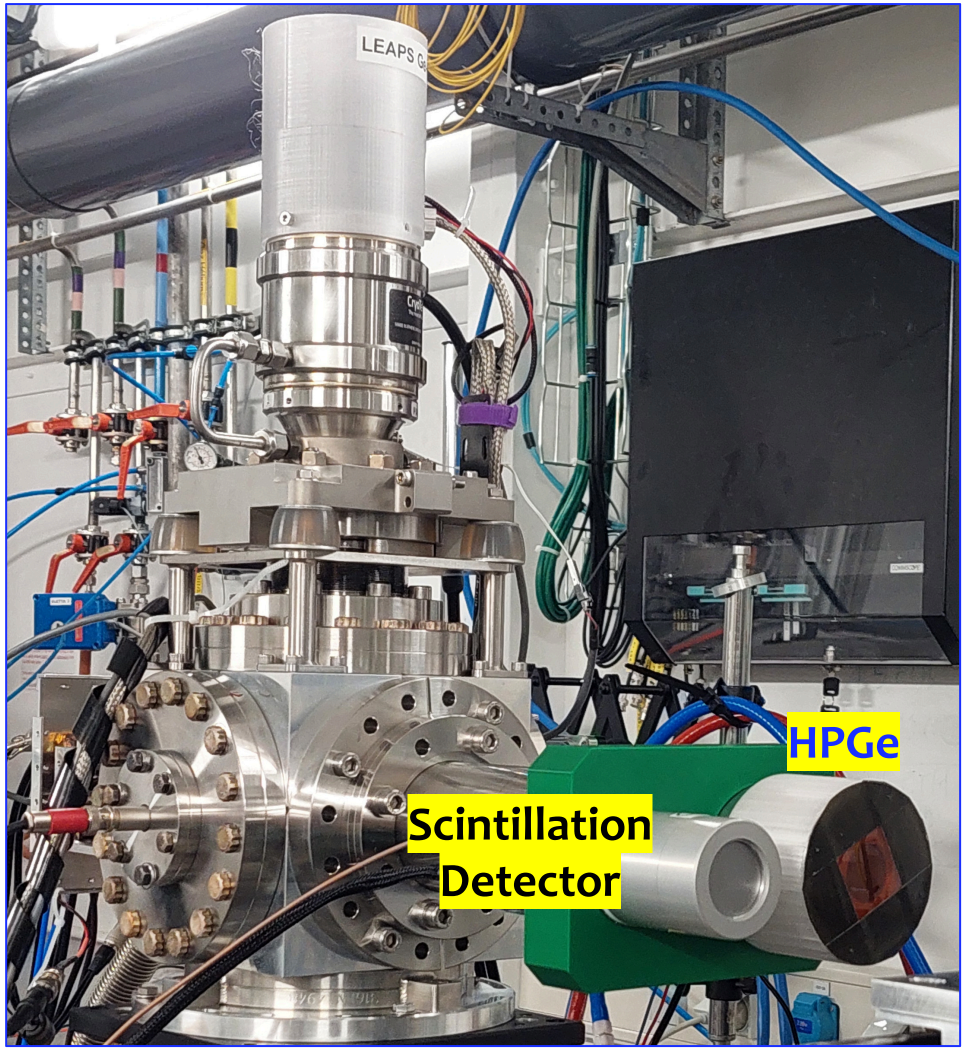
May 2025 ESRF- EBS BM05

- **Scans across the whole sensor surface at different energies**(20,30,40, and 50 keV), Pixel map +Energy spectra
- **Low- and high-resolution scans over some pixels with increasing bias voltage** (from 70 V to 250V)
- **Quantum efficiency measurements using as a reference a photon-counting detector.**
- **Evaluating Dead Time by varying photon flux** (ICR vs. OCR)
- **Acquiring XRF spectra at 50 keV** with Sb, Ag, Cs, CsI, GdTb, and EnviroMAT soil samples.

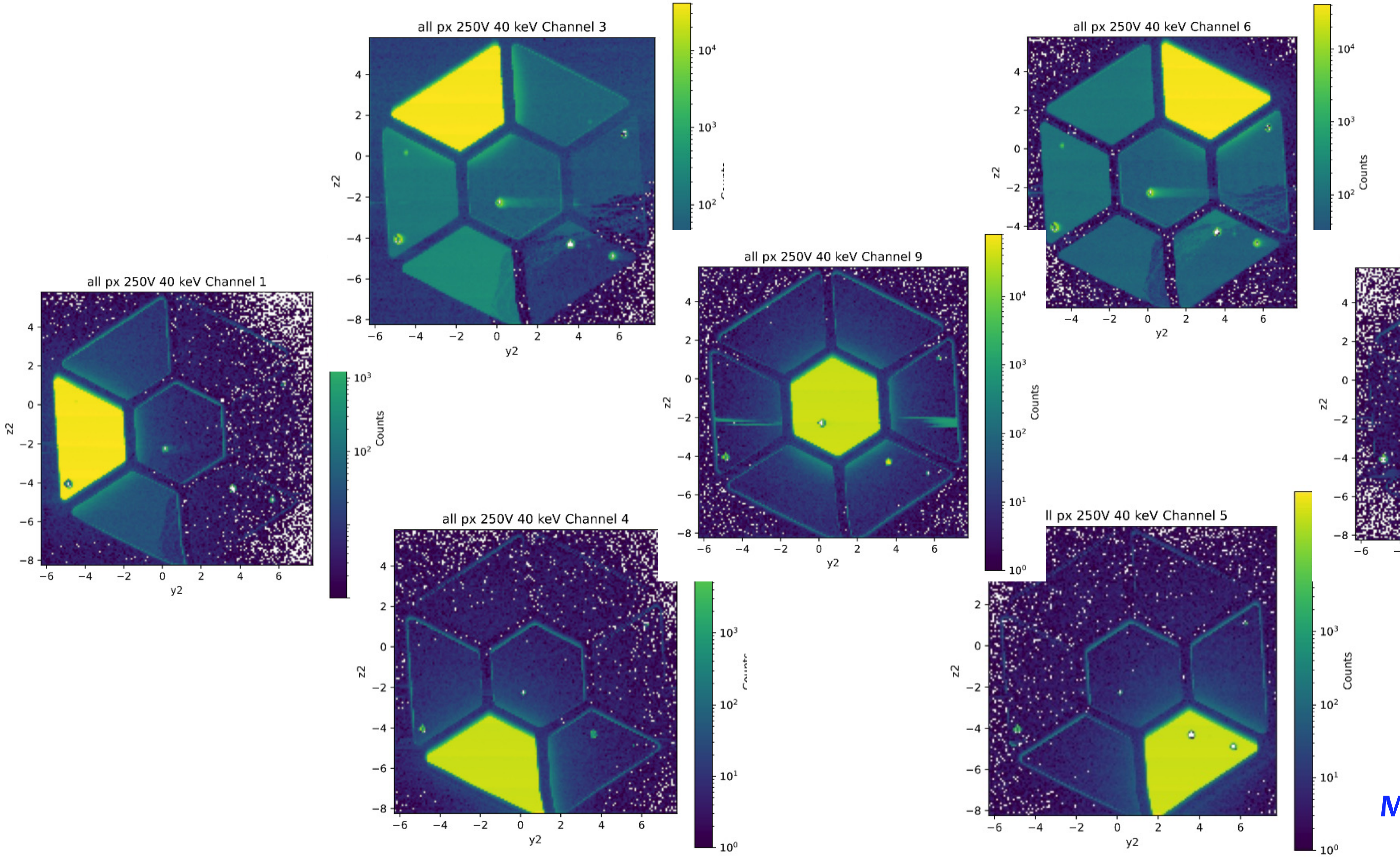
Data analysis is on going

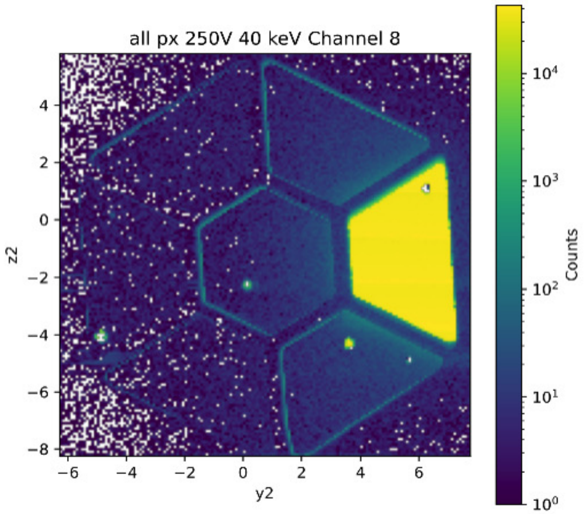
XAFS- DET at the BM05 beamline





Scans across the whole sensor surface

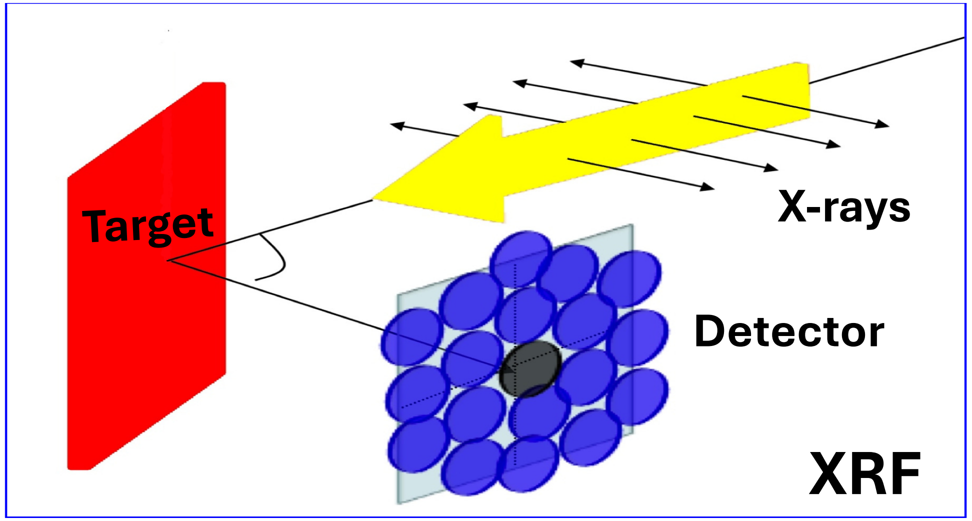


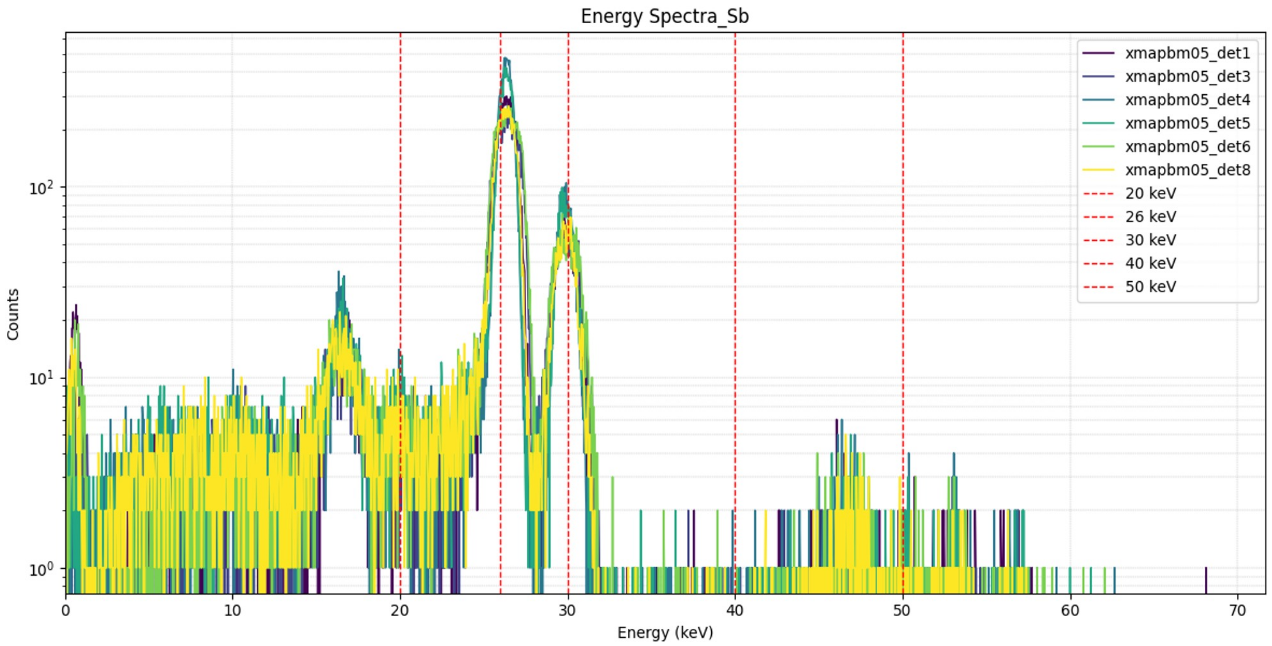


1) 30 µm X-ray beam
2) 40 keV
3) V_B=250 V

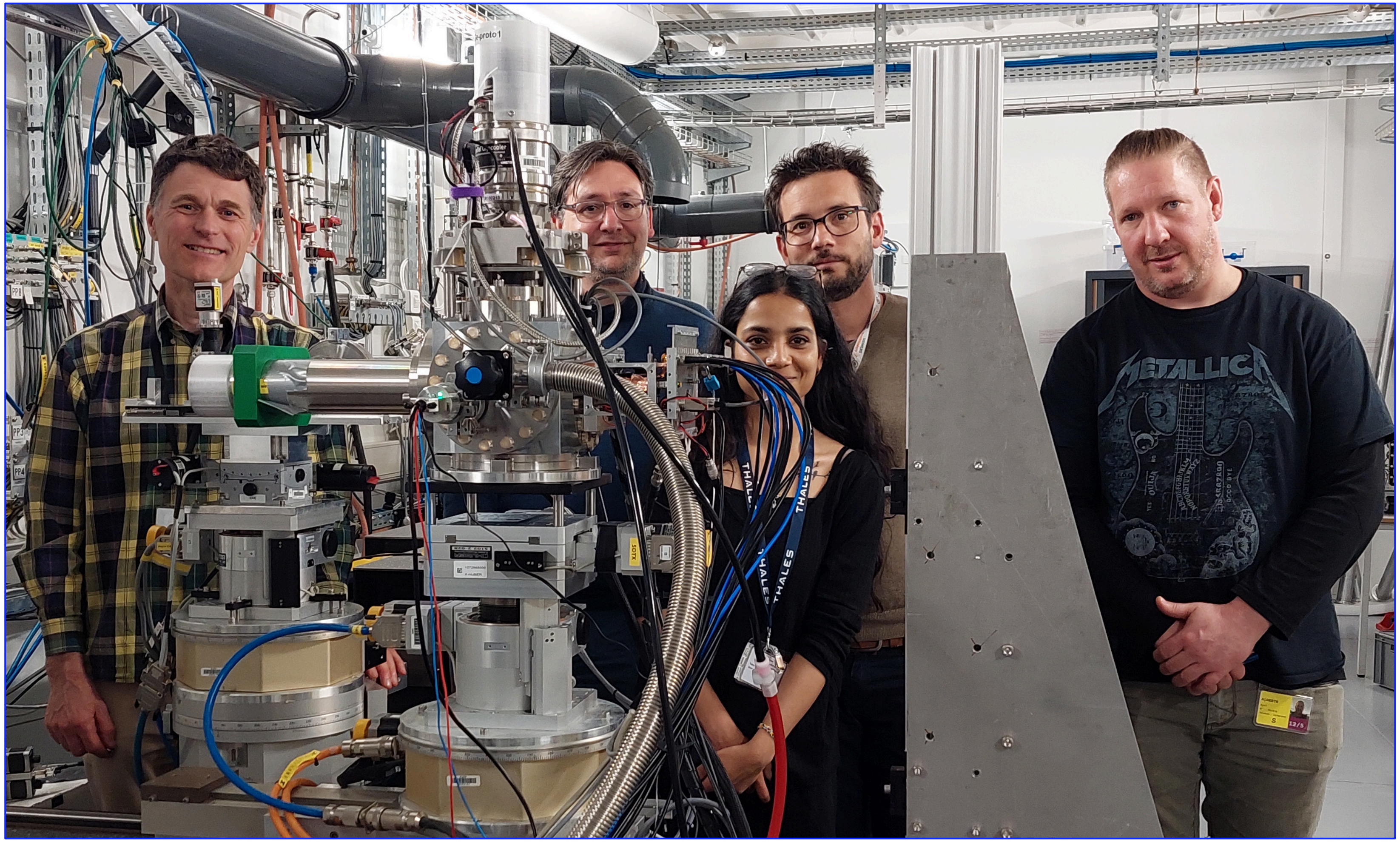
M. Cascella - MAX IV

First SR XRF spectra





End of detector tests May 2025 at ESRF-EBS BM05



This first SR campaign identified the need for further optimization, specifically in reducing system noise and enhancing electronic stability to support long-duration, high-count-rate experiments.

Conclusions

Sharing knowledge can produce important results.

Acknowledgments: This project has received **funding from the European Union's Horizon 2020** research and innovation programme under **grant agreement N. 101004728**.

Publications

1. **LEAPS-INNOV XAFS-DET Consortium**, "*Conceptual Design Report for the germanium detector prototype system*" Research and Innovation Action (RIA) under the Grant Agreement no 101004728, Deliverable no. D2.1, 21 September **2021**.
2. **F. Orsini**, S. Aplin, A. Balerna, P. Bell, J. Casas, M. Cascella et al., *Xafs-det: A new high throughout x-ray spectroscopy detector system developed for synchrotron applications*, Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment 1045 (**2023**) 167600
3. **M. Quispe** et al., "*Thermal Mechanical Simulations of a New Germanium Detector Developed in the European Project LEAPS-INNOV for X-Ray Spectroscopy Applications at Synchrotron Facilities*", in Proc. IPAC'23, Venezia, Italy, May. **2023**, pp. 4389-4392. doi:10.18429/JACoW-IPAC2023-THPA18
4. **L. Manzanillas**, S. Aplin, A. Balerna, P. Bell, J. Casas, M. Cascella et al., *Development of multi-element monolithic germanium detectors for x-ray detection at synchrotron facilities*, Nuclear Instruments and Methods in Physics Research Section A: Accelerators, Spectrometers, Detectors and Associated Equipment 1047 (**2023**) 167904
5. **M. Quispe**, "*Germanium Detector: Thermal and Vibrational Studies*", LEAPS-INNOV WP2: XAFS-DET. Annual Meeting, Krakow, Poland, April **2024**.
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7. **N. Goyal** et al. *Progress in the Development of Multi-Element Monolithic Germanium Detectors in LEAPS-INNOV Project: Insights from Detector Performance Simulation.* Journal of Physics: Conf. Series, **2025**, 3010, 012120
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Thank you for your attention