MIRACLES
STAP Spectroscopy

José E.M. Pereira & Félix J Villacorta,
on behalf the MIRACLES Team
23/10/2024

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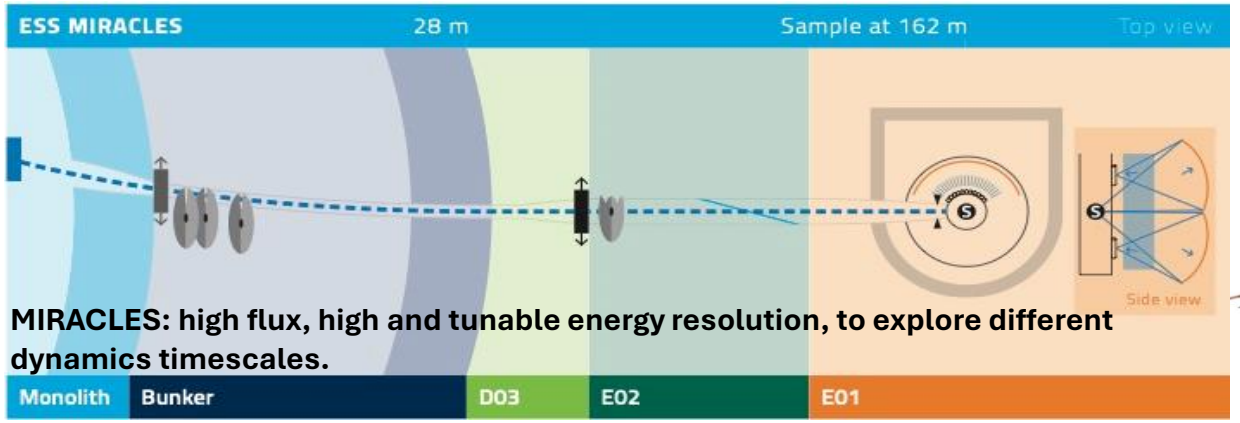
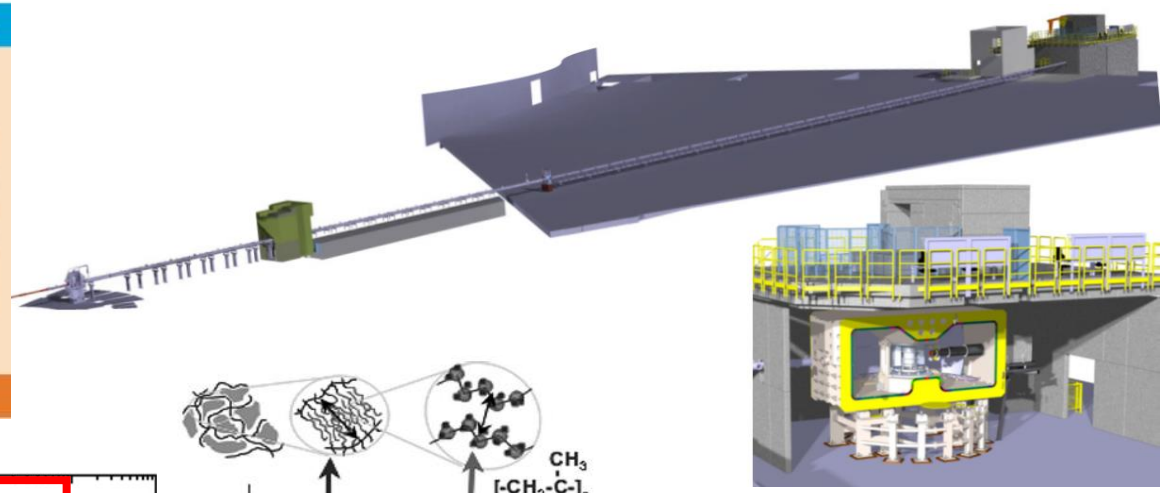
GOBIERNO DE ESPAÑA MINISTERIO DE CIENCIA E INNOVACIÓN
EUROPEAN SPALLATION SOURCE

MIRACLES, the TOF Backscattering Spectrometer

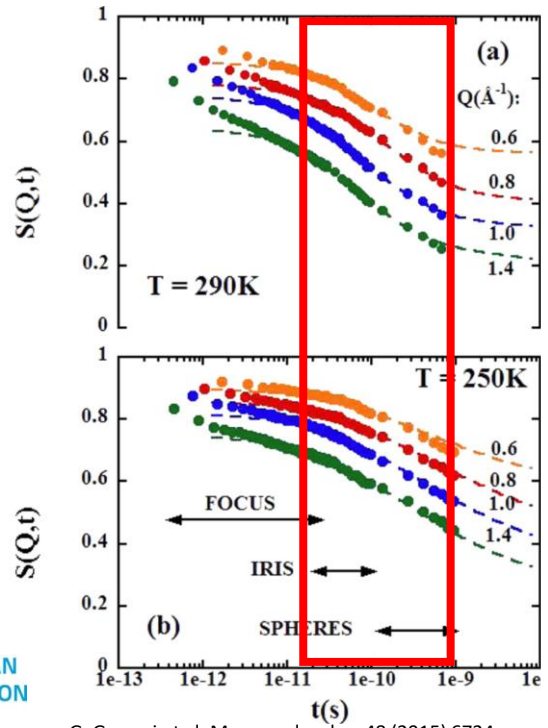


MIRACLES: high flux, high and tunable energy resolution, to explore different dynamics timescales.

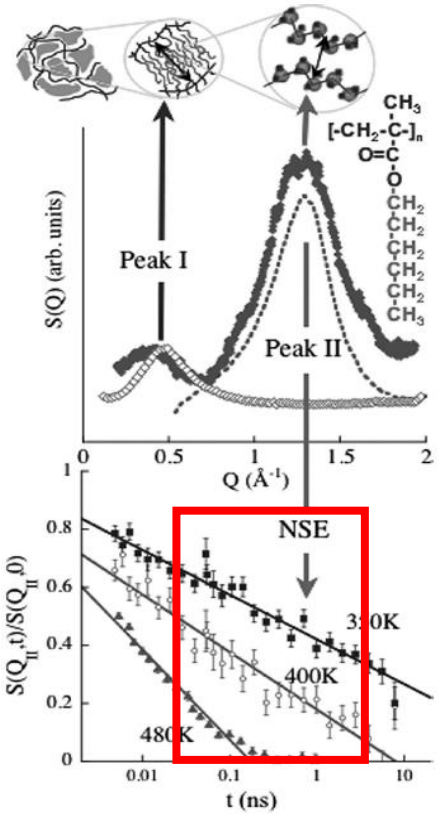


Different timescales, one instrument!

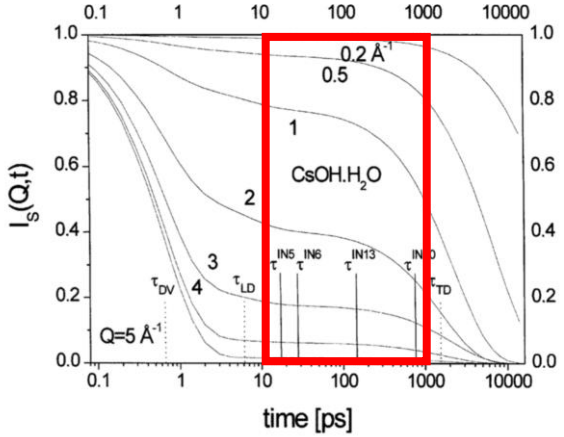
- **The possibility of carrying out one experiment at different timescales (ns-ps).**
- **Chopper cascade to select the energy resolution in QENS measurements.**



G. Goracci et al. Macromolecules. 48 (2015) 6724-6735



Arantxa Arbe & Juan Colmenero (2010), Neutron News, 21:3, 11-14



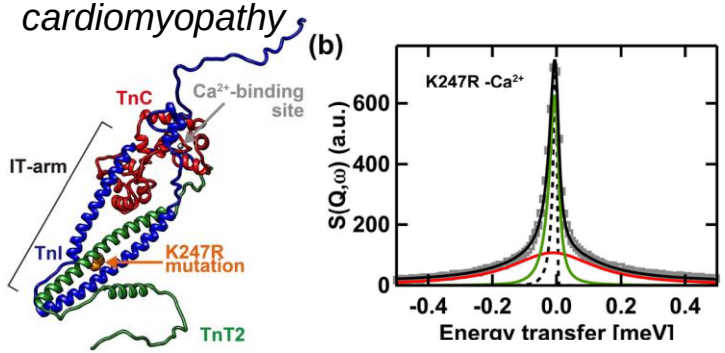
R. E. Lechner, Physica B 301 (2001) 83–93

MIRACLES Science Case

Life science

Dynamics of Proteins and Water

Example: Dynamics of troponin in cardiomyopathy

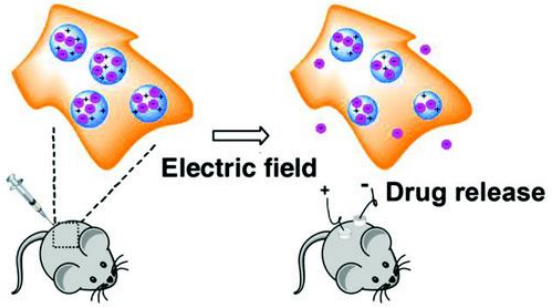


T. Matsuo et al., BBA-Proteins and Proteomics, 1865, 1781 (2017)

Pharmaceutical science

Drug Delivery

Example: drug release from electric-field responsive nanoparticles

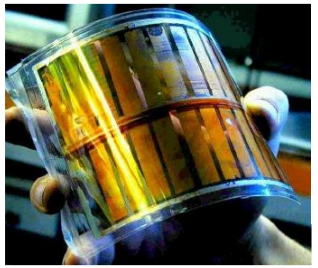


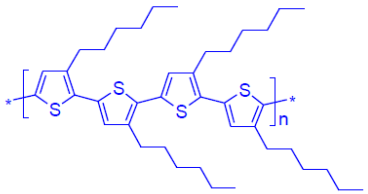
J. Ge et al., ACS Nano. 6, 227-233 (2012).

Polymer science

Morphology-performance connections

Example: Polymers in organic photovoltaic devices



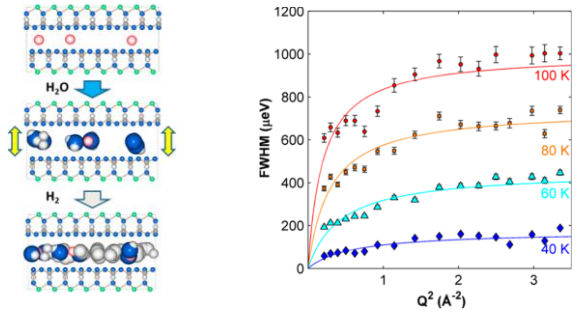


G. Paternó et al.,Chem. Phys.427, 142 (2013)

Energy science

Hydrogen Storage

Example: Hydrogen adsoprtion in MOF

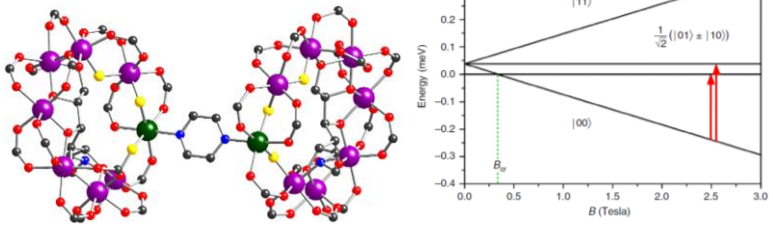


J.S. Edge et al., J. Phys. Chem. C, 118, 25740 (2014)

21st century Magnetism

Quantum Information Processing

Example: Molecular magnets and quantum entanglement

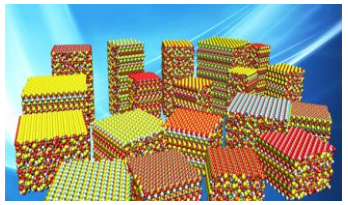


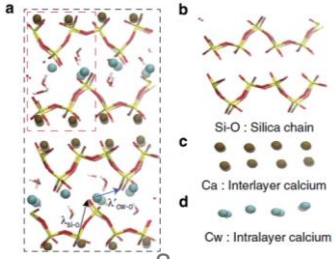
E. Garlatti et al., Nature Commun. 8, 14543 (2017)

Environment science

Greener building materials

Example: use of greener cements to reduce greenhouse gases





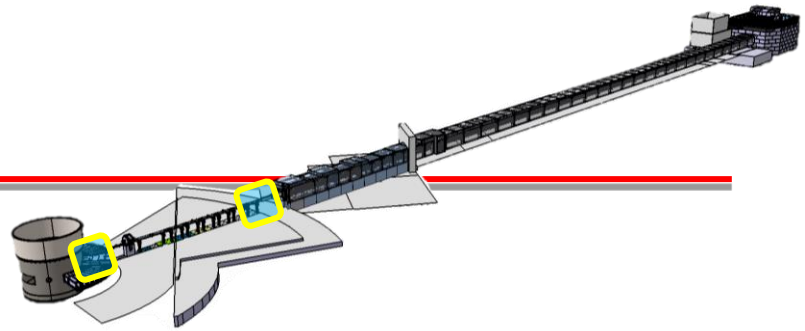
J. Jacobsen et al., Sci. Reports 3, 2667 (2013)

Status and management

The MIRACLES instrument has started installation of the first components, the rest of the components are in the manufacturing phase (being manufactured, manufactured and with FAT tests ongoing or ready to be installed).

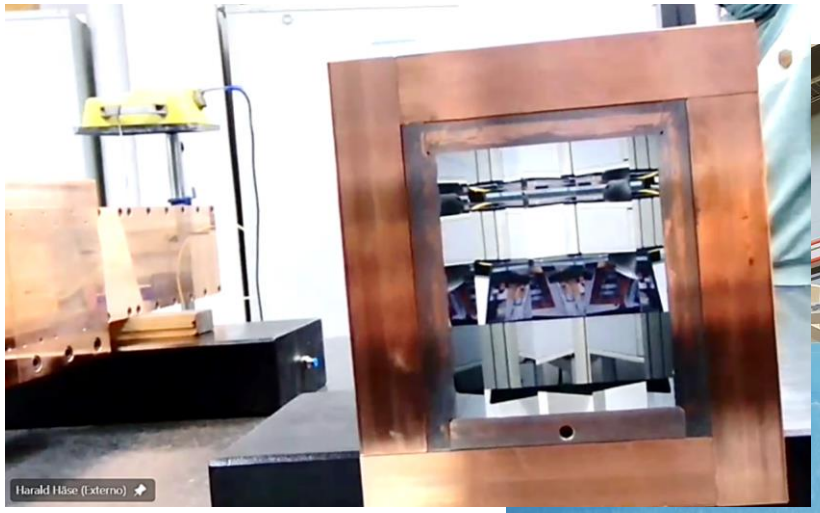
- Installed:
  - **NBOA(2021)**
  - **BBGOA**
  - **In-bunker guide**
  - **In-bunker guide supports**
  - **BWI**
  - **Control room**
- Manufactured (FAT ongoing or approved):
  - Out-of-bunker guide
  - D03 out-of-bunker guide supports
  - Scattering vessel
  - Choppers
  - Detectors electronics (front end)
- Manufacturing:
  - E02 & E01 Out-of-bunker guide supports
  - Analyzer
  - Detectors
  - Radial collimator
  - Get-lost tube/Beam stop
- Detailed design:
  - Cave
  - Sample area equipment
  - Slit

Beamline feedthroughs

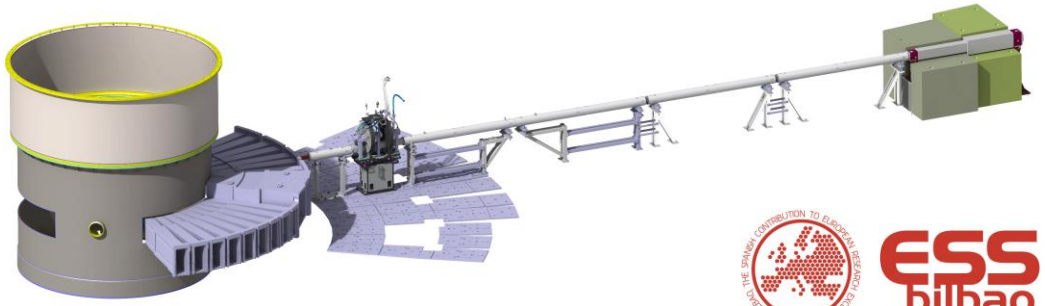


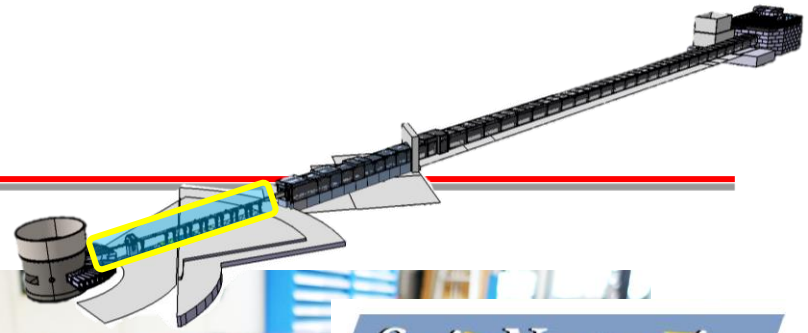
Neutron Beam Extraction (NBOA)

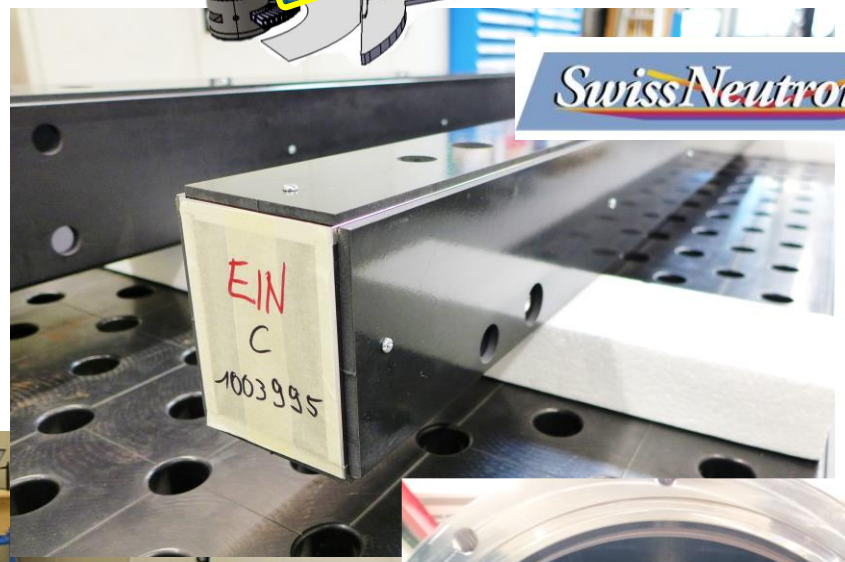
Bunker Wall Insert (BWI)

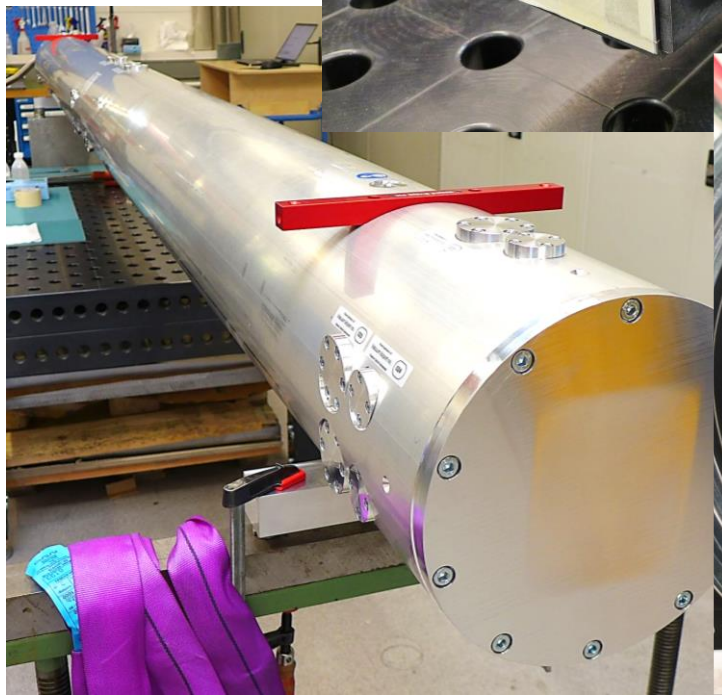


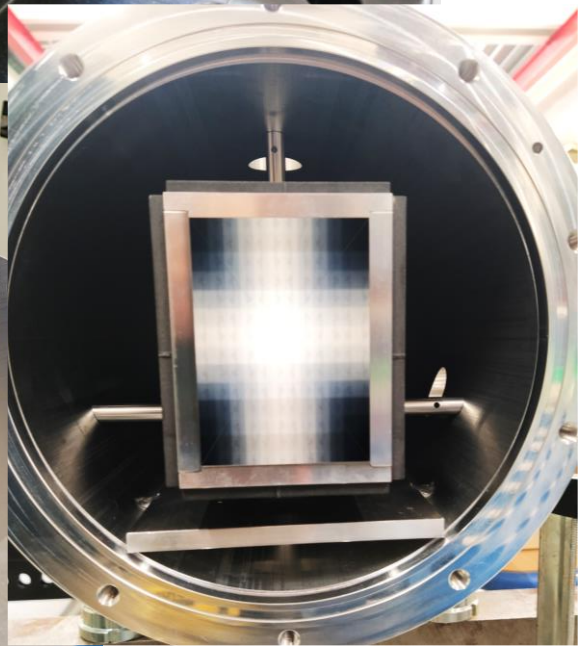
In-bunker neutron guides





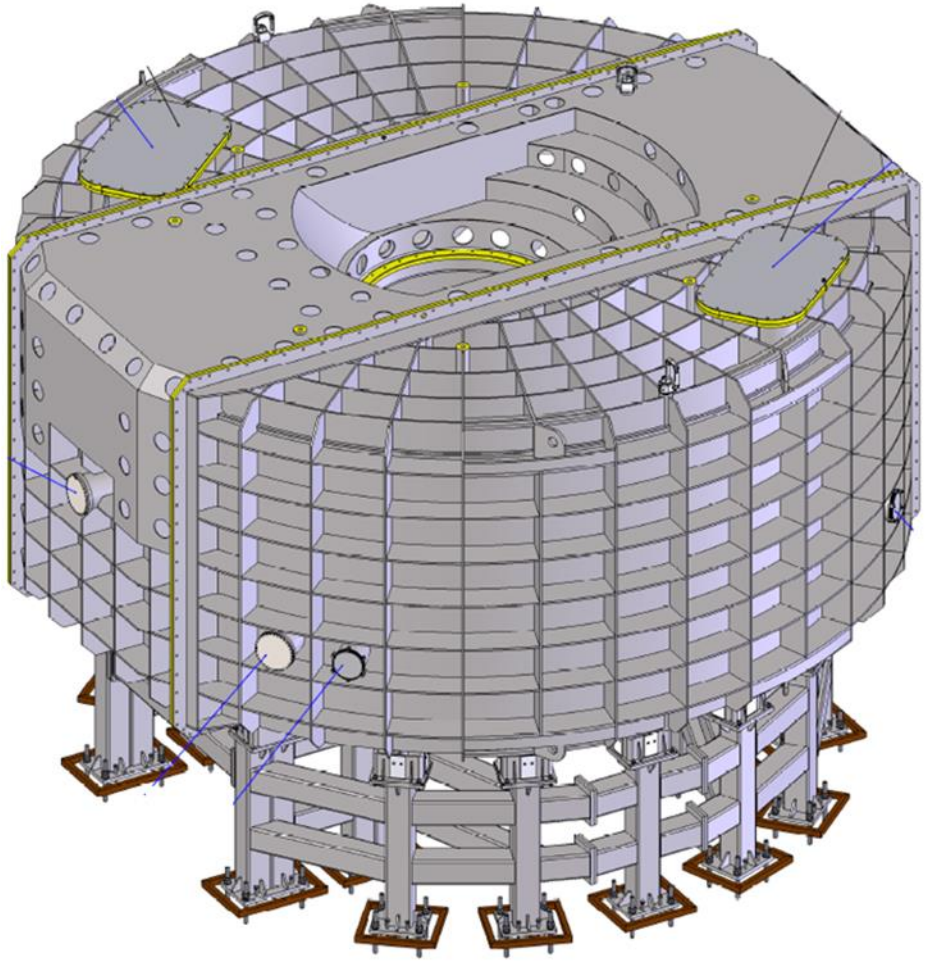






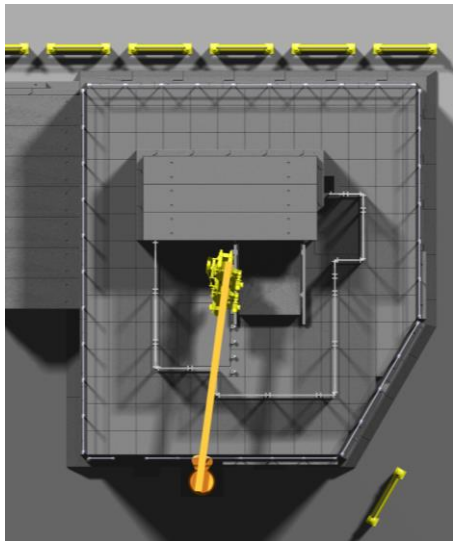
Scattering vessel

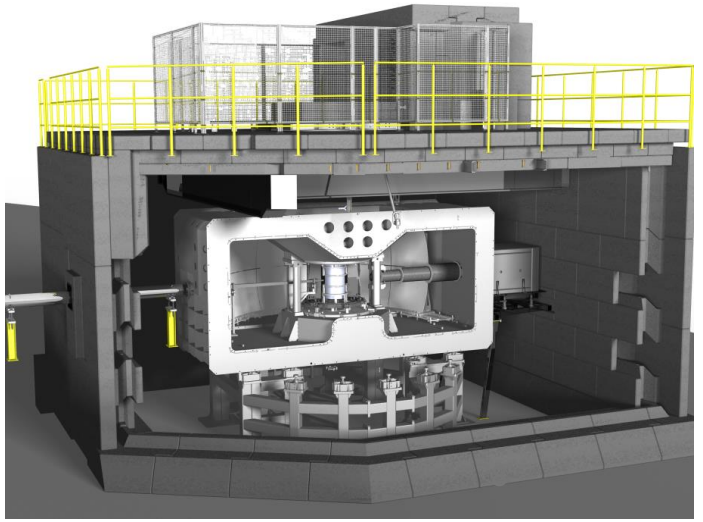
- ✓ Deflection at sample position: ≤ 1.5 mm (vertical)
- ✓ Steel 316L (in agreement with Polarization Analysis)
- ✓ Vacuum ~10^{-3} mbar



Experimental end station

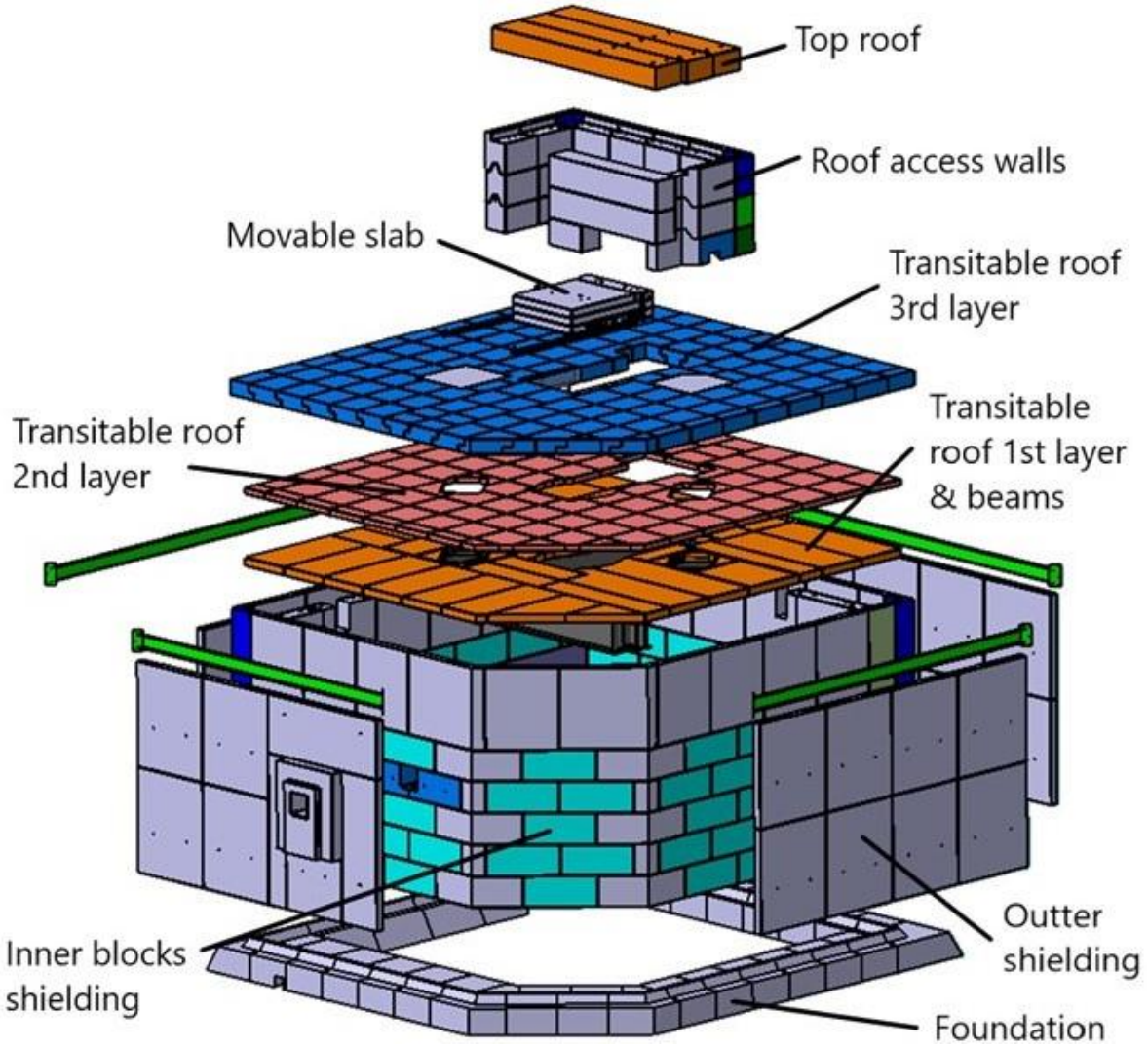
Experimental cave: the main building for scientific activities (roof), giving access to the sample position, hosting the sample preparation areas and handling / moving sample environment equipment.

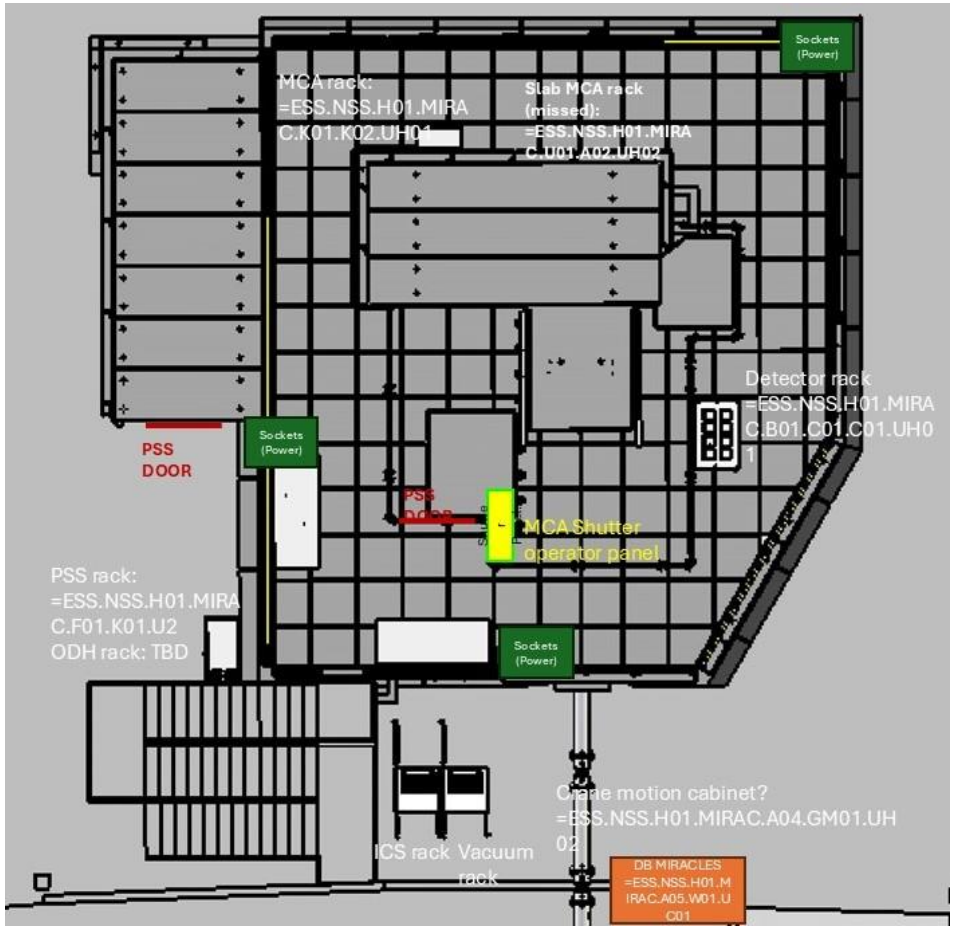




Experimental end station

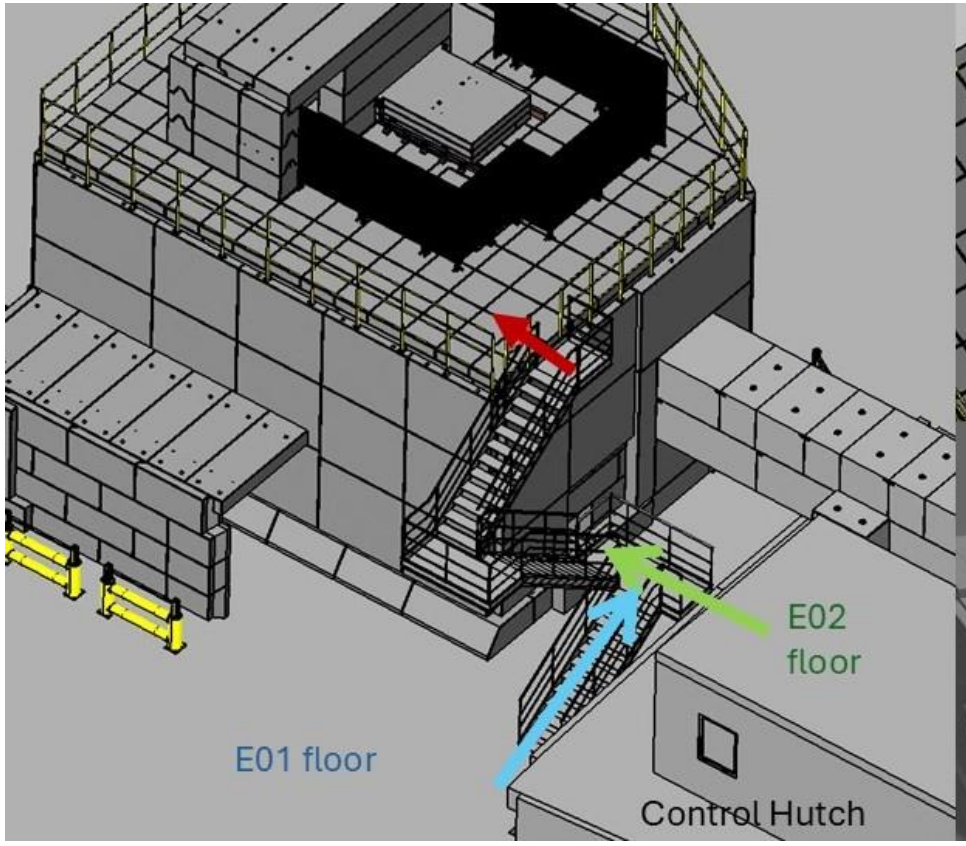
Cave design completed with the integration of the utilities, also considering development and planning related to operational aspects.

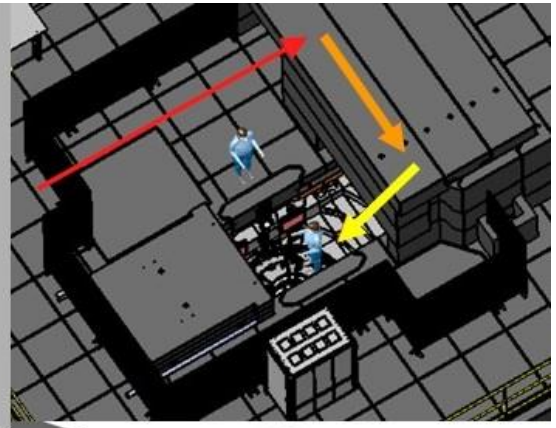


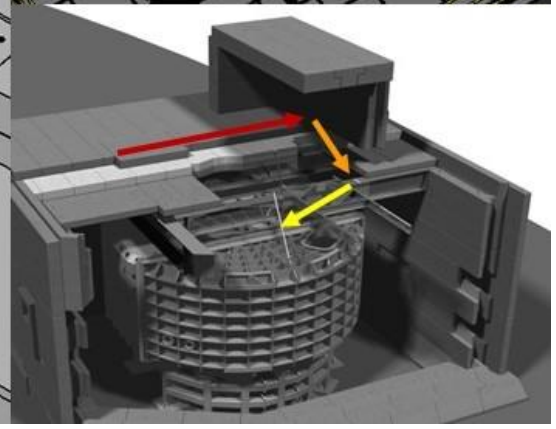


Experimental end station

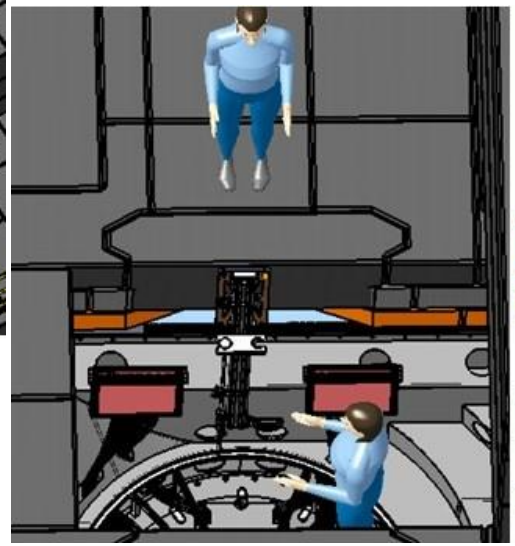
- Access to the sample position.
- Hosting the sample preparation areas and handling.
- Moving sample environment equipment.

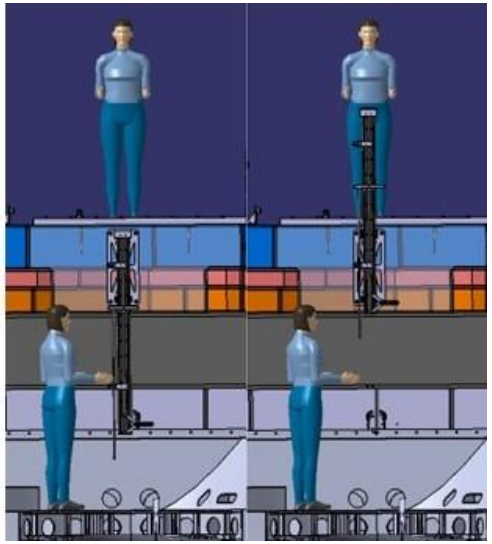


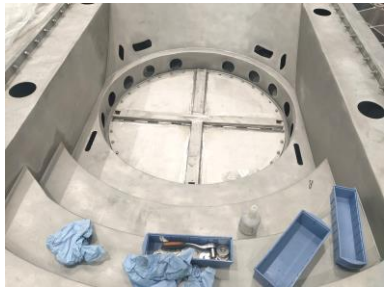




A stick holder will be attached to the roof of the cave blocks to allow to reach the sticks for ergonomic transfer from and to the cave's roof to allow a fast release of activated samples.

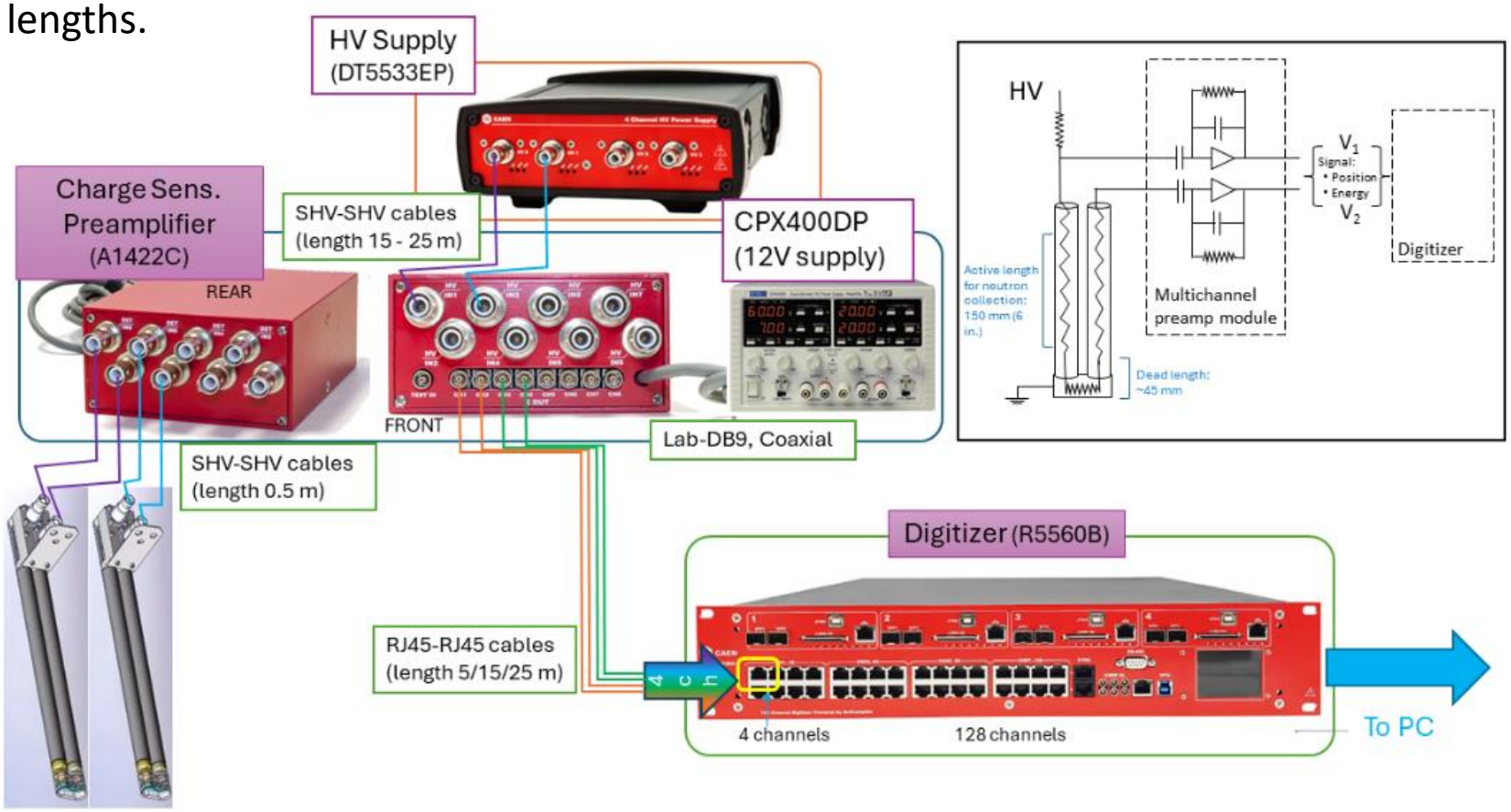


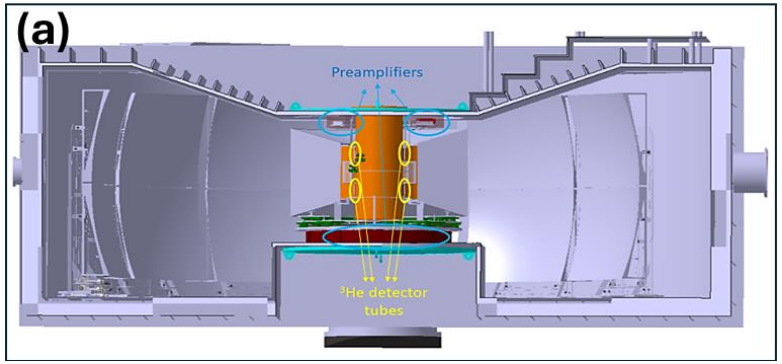


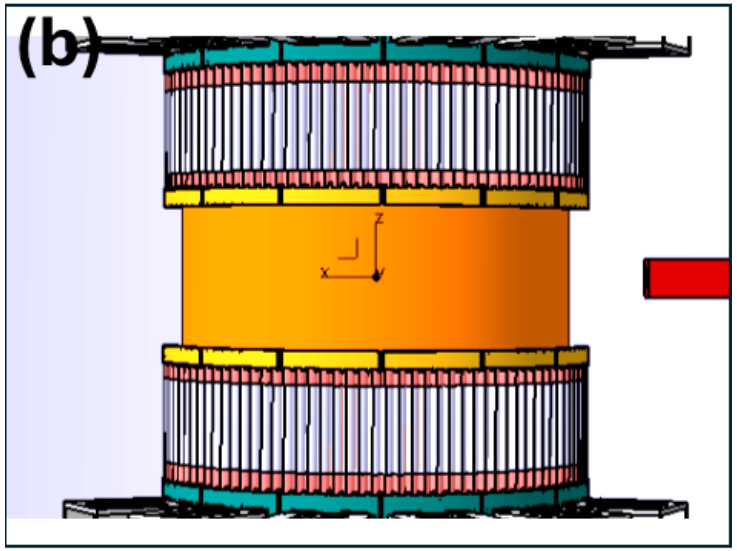


Detectors & DAQ chain

A doublet assembly formed by two ^{3}He tubes were design and manufactured to check the robustness of the U-shaped connection in terms of thermal stability, ground and RF isolation; two preamplifier modules were also tested. Finally, integration tests between the electronic devices included different cable lengths.



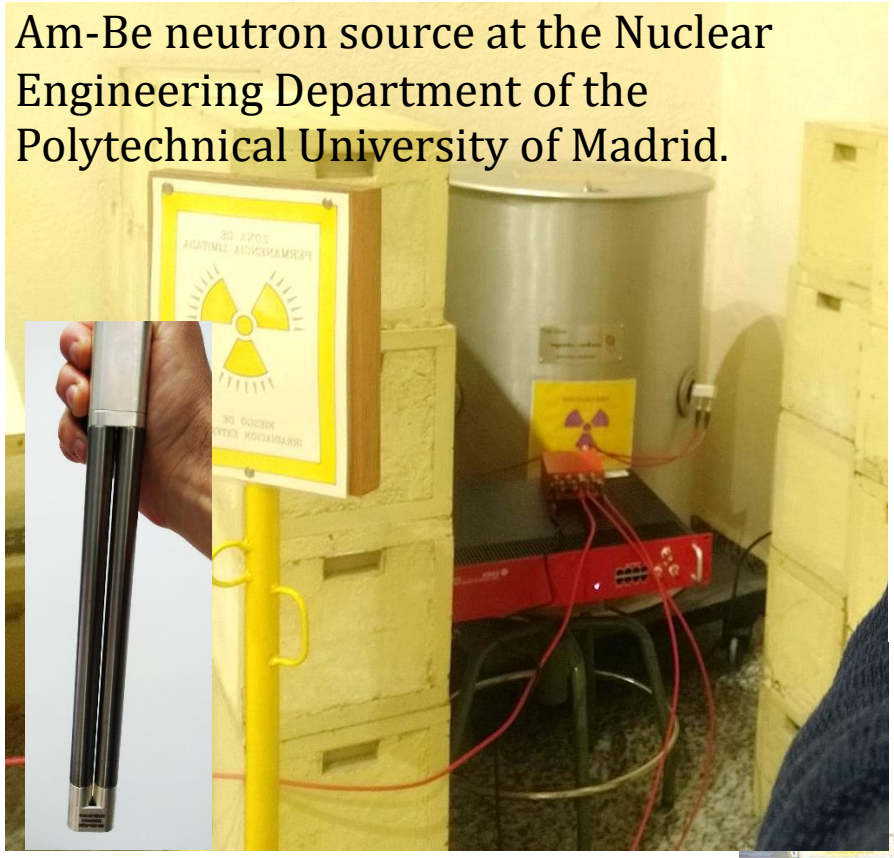


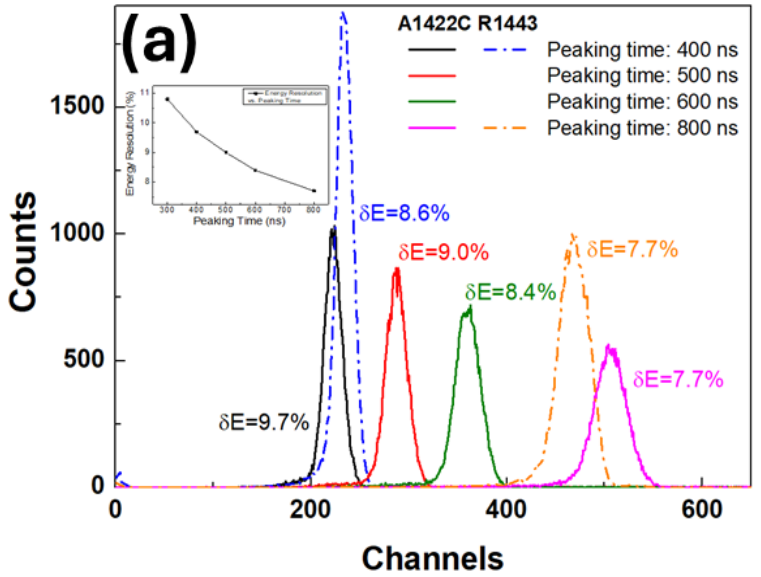


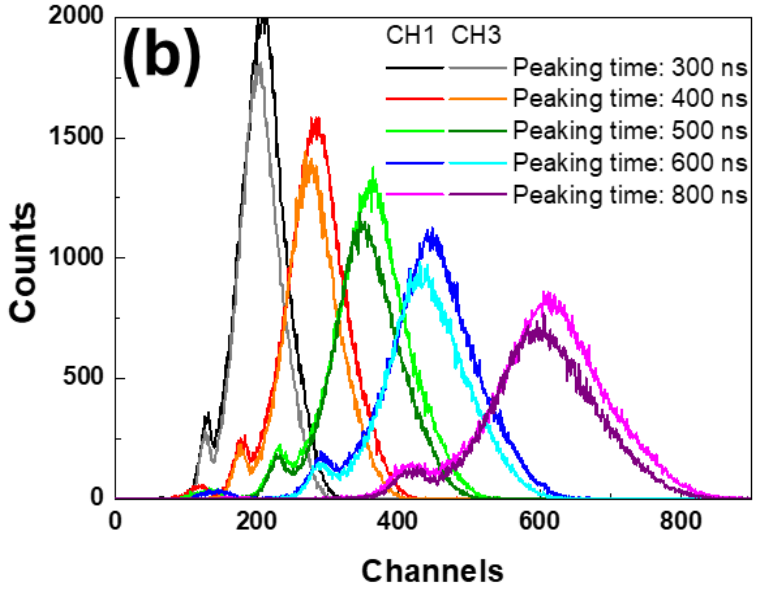
I. Mazkiaran, et al., *submitted*

Detectors & DAQ chain

Am-Be neutron source at the Nuclear Engineering Department of the Polytechnical University of Madrid.







Pulsed height spectra vs peaking time using the A1422 preamp module as a function of peaking time.

(a) in the counter configuration, including results from the R1443 preamp for comparison (inset: evolution of the energy resolution with peaking time);

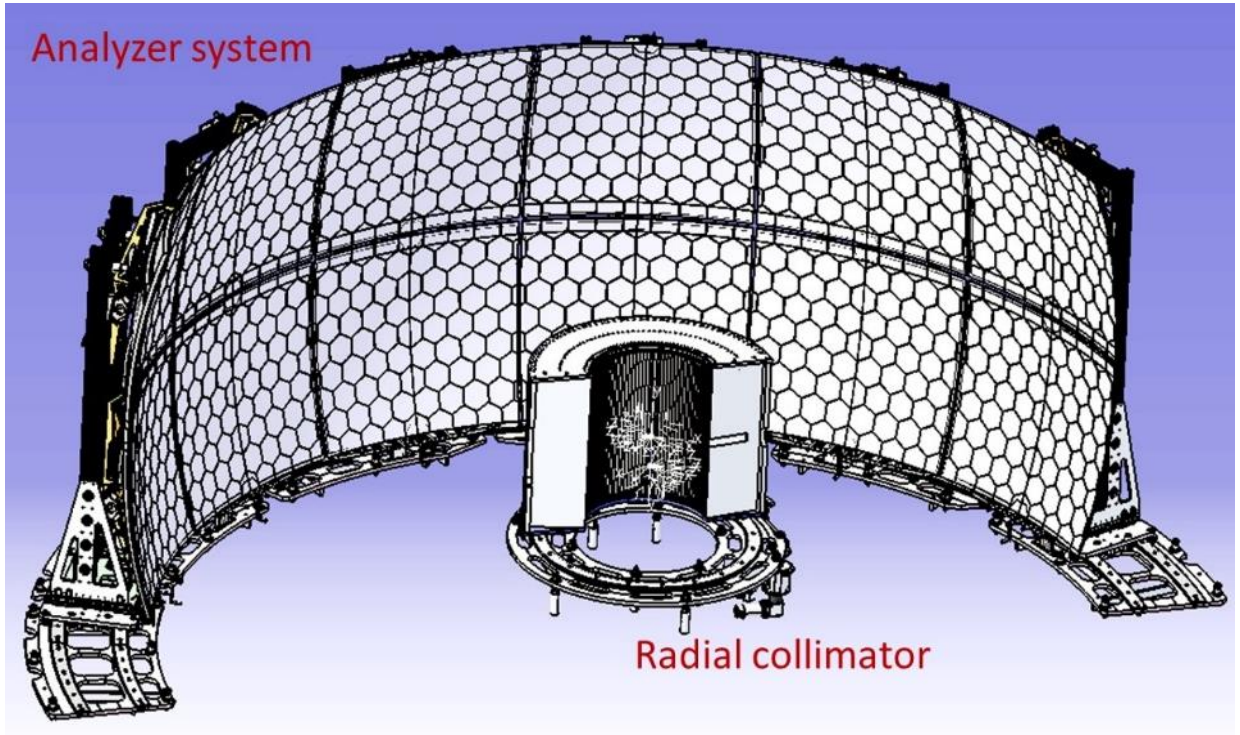
(b) in the position sensitive configuration.

I. Mazkiaran, et al., *submitted*

Analyzer system

MIRACLES analyzer consists of:

- 2-spherical monochromator of radius 2.5 m.
- 12 spherical plates (6 plates for the top set and 6 for the bottom set).
- >1000 Si(111) wafers, 6 in. diameter, cut into hexagons.



The performance of the spectrometer, in terms of **energy resolution and flux, depends** critically on the features and the assembly procedure of the **Si(111) crystals**.

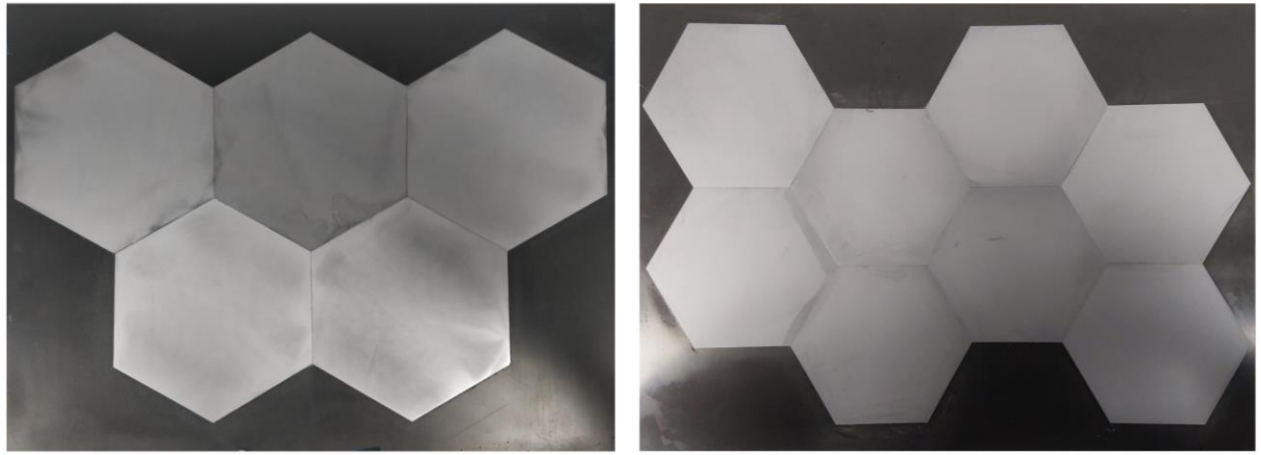
A systematic analysis of Si(111) wafers with different parameters was performed using the backscattering spectrometer IN16B at the Institut Laue-Langevin (ILL).

Prototype analyzer

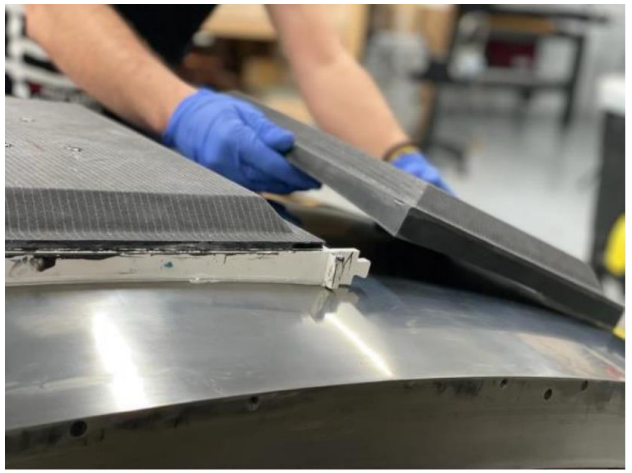
For the experiment, 10 panels distributed in 2 prototypes mounted with different Si crystals samples each, distributed into distinguished areas, onto spherical surface with a radius of 2000 mm.

Si samples:

- Single side polished.
- Hexagonal shape: 6 inches (≈150 mm).
- Thickness: 575, 750, 1000, 1250 mm.
- Surface: etched (EC) *vs.* as-ground(AG).
- Growth method: Czochralski(CZ) *vs.* float zone(FZ).

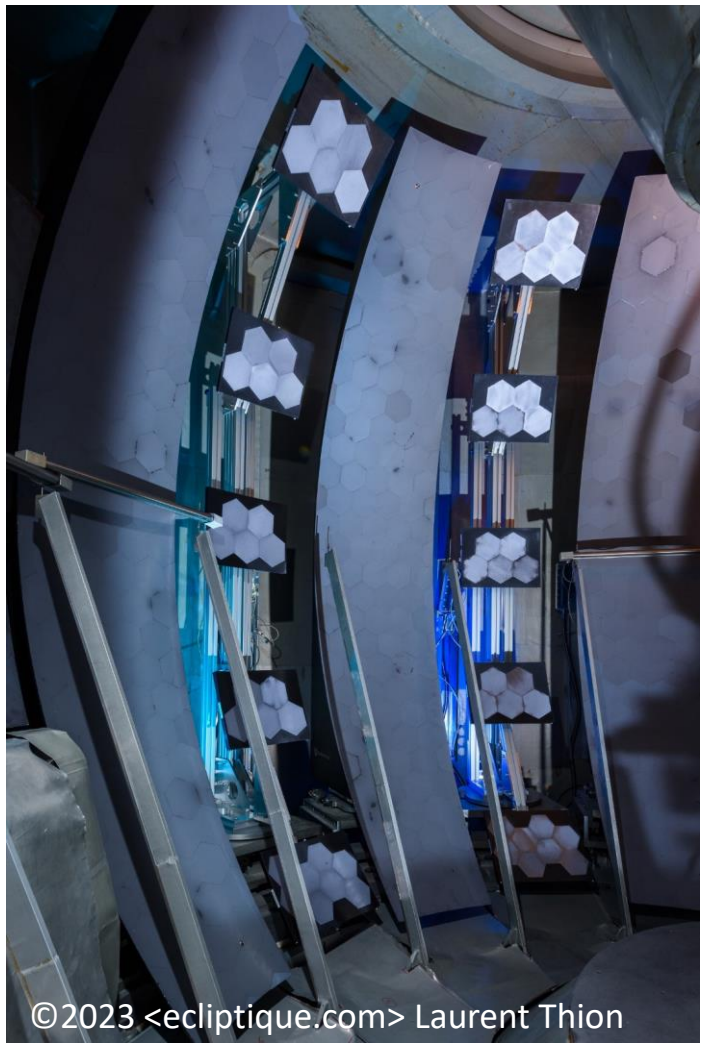


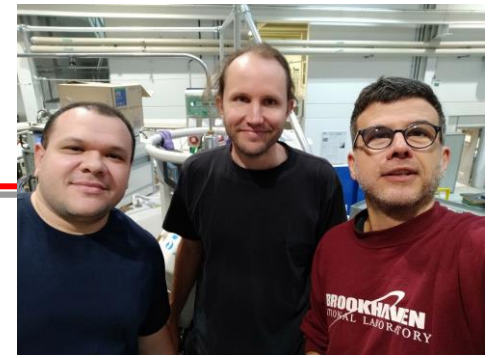
Si hexagonal with 5 inches (~120 mm) and samples with 6 inches (~150 mm), the area covered with Si are the same ≈ 0.07m^2.

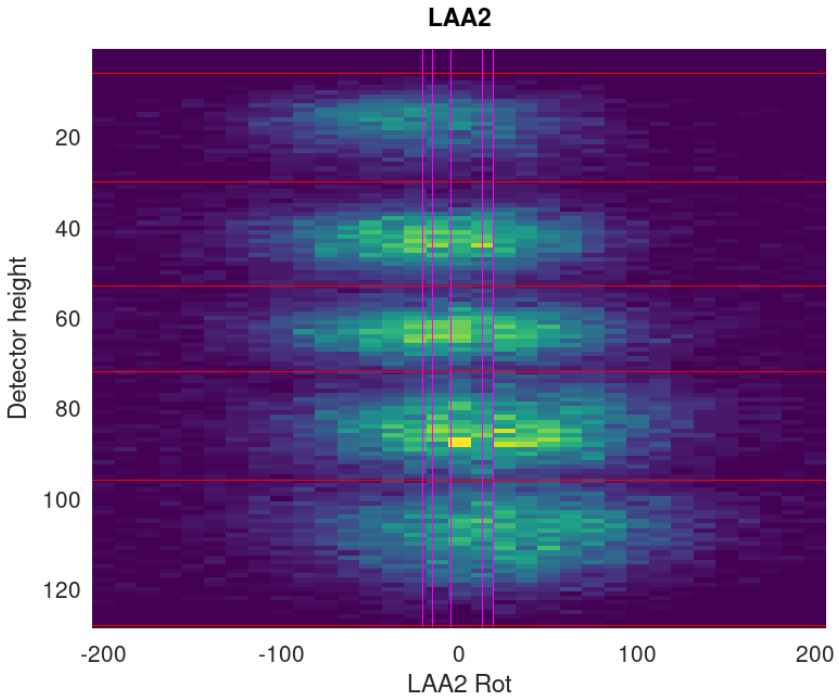


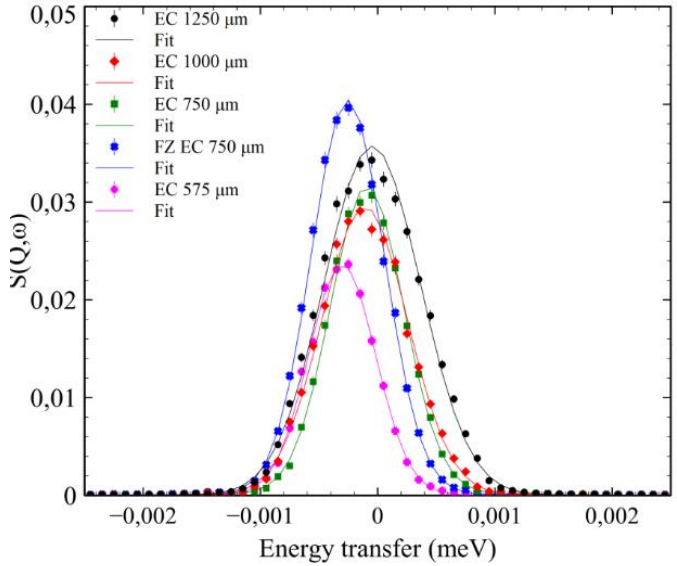
Si crystals glued using LOCTITE® EA 9466™.

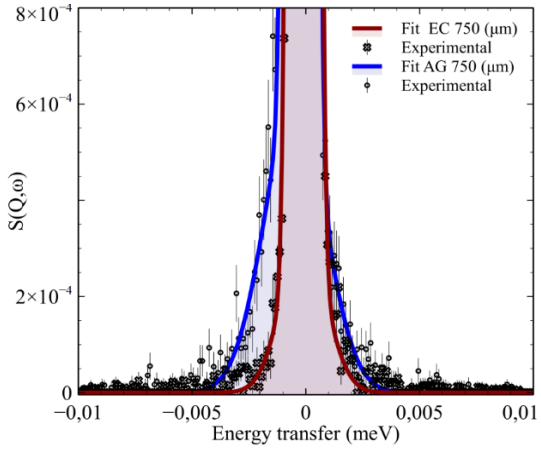
Prototype analyzer

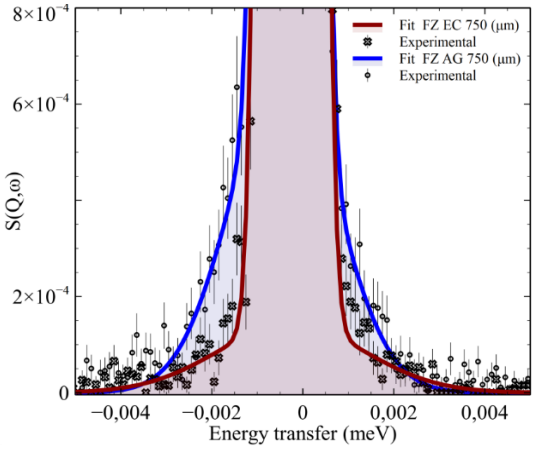


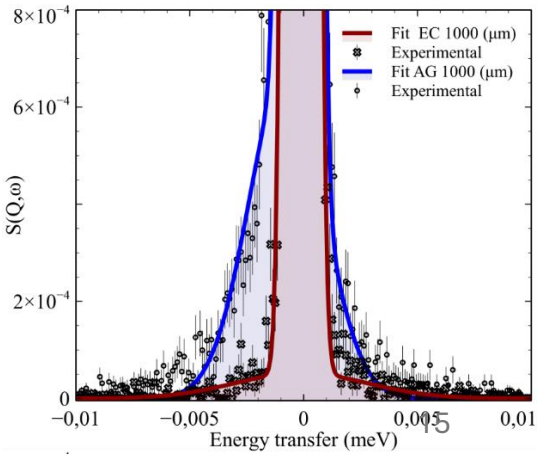




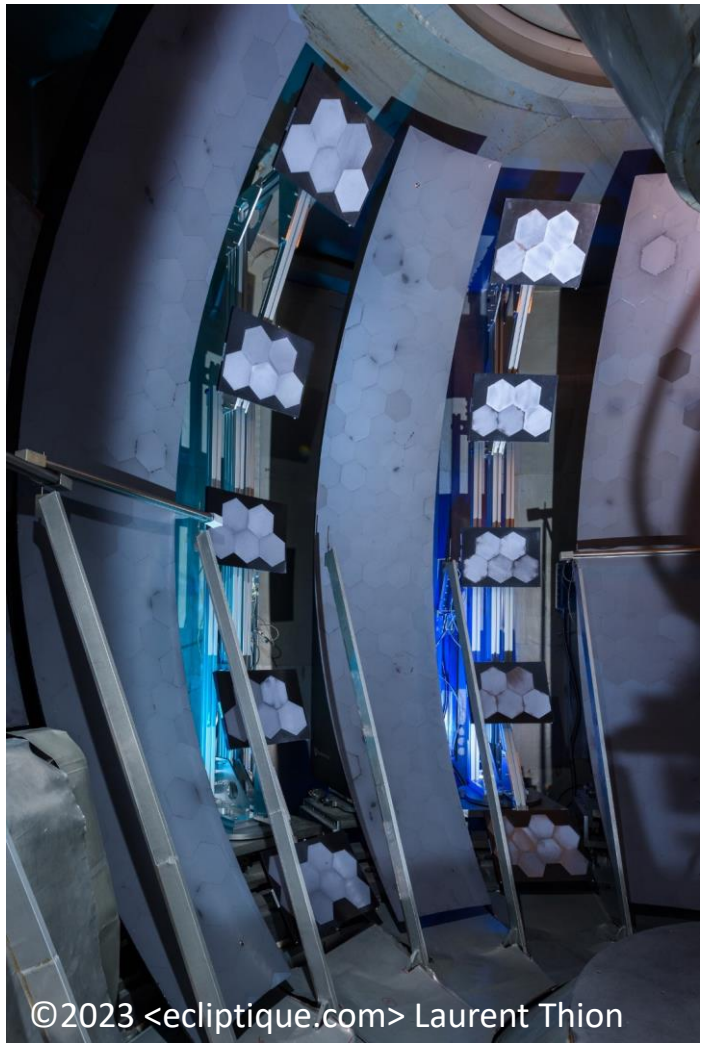




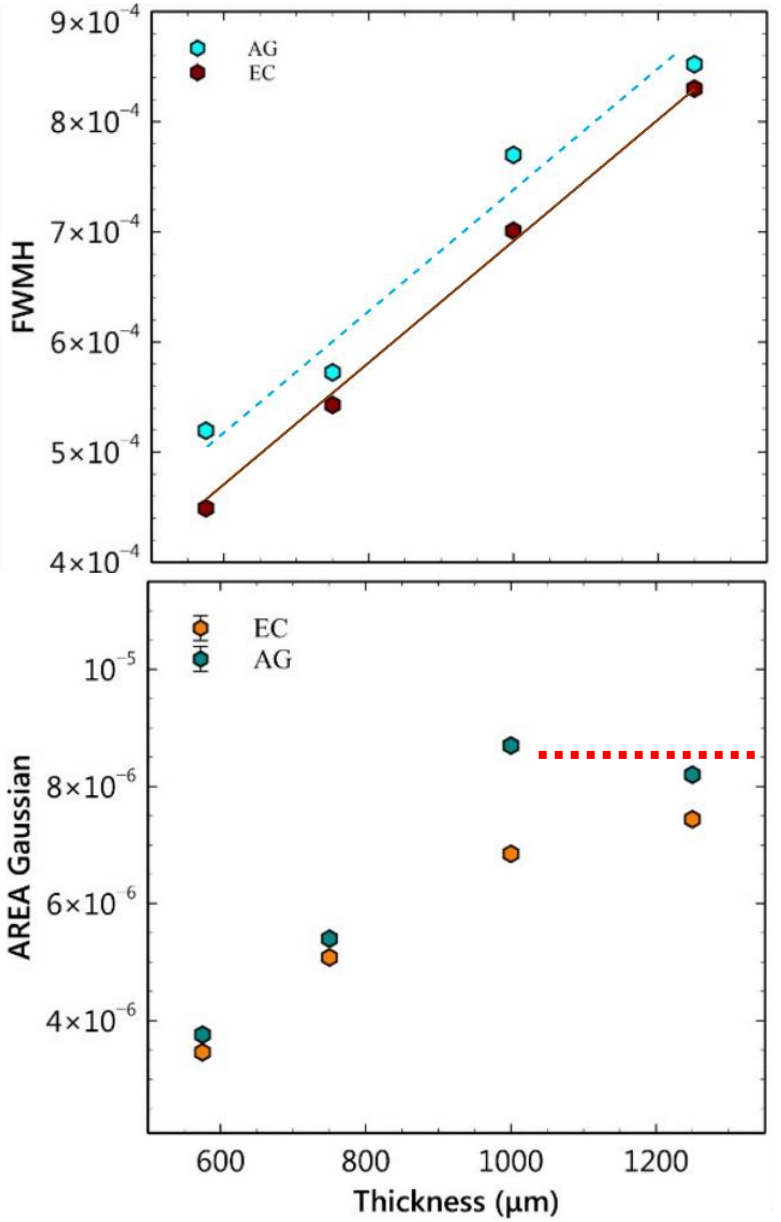




Prototype analyzer



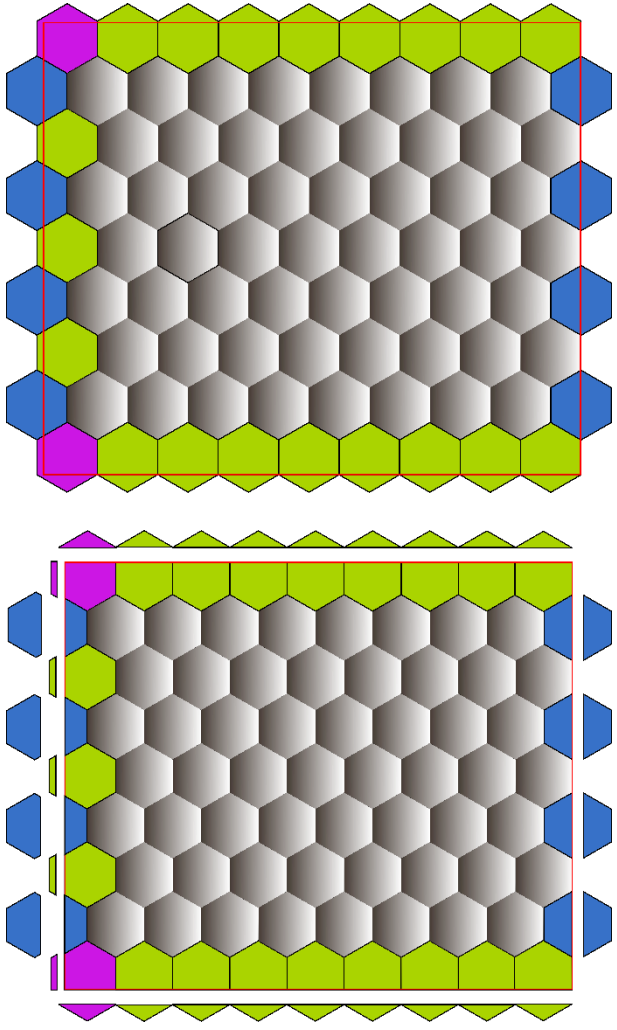
- Flux FZ > CZ.
- EC crystals show symmetric QENS. So, we select the EC ones.
- Increasing thickness does not convey a relevant increase in flux (i.e. starts to reach the maximum limit).
- Saturate around 1000 µm.
- FZ 1000 µm EC seems the most suitable choice for MIRACLES.

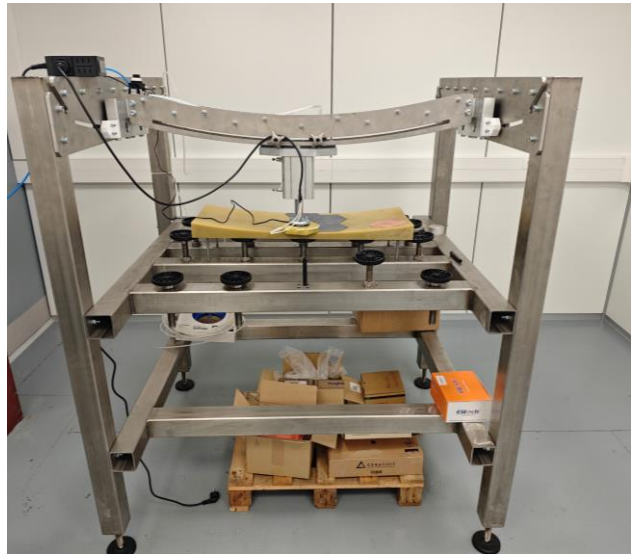


1º batch of the Si delivered!

MIRACLES analyzer optical assembly

85 pieces (full hexagon or fraction) are needed for to cover one panel. 1020 for the total analyzer system.





Station for bending and gluing the Si crystals; pneumatic press; stamp head with a heating wire for keep temperature constant during the curing process of the glue.

Sample positioning system

3-sample linear changer

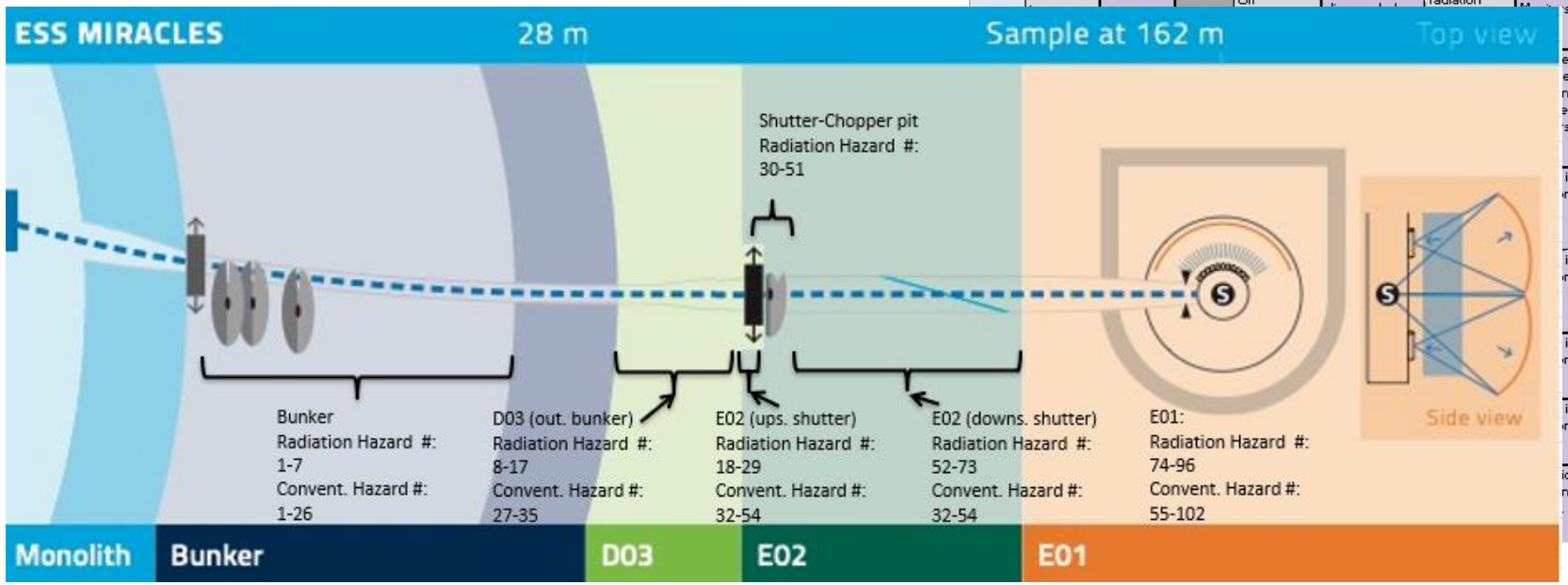
- Loading up to 3 samples, in agreement with the Z-positioning range.
- Adaptor from treaded connection (sample stick) to bayonet connection.
- Lower ends with bayonet connection for interchangeable order, and also to make a fast release to the lead box.

Bayonet-like connection for fast release

IHA workshop

- ODH (MIRACLES ODH assessment ESS-5487918).
- Radiation, conventional hazards and their mitigations.

	Radiation Hazard Identification								Risk Estimation and Reduction			
	1		2	3	4	5	8	9	10	11	12	13
Building	Instrument Area	Instrument Sub-area	Radiation Hazard Number	Mode	Cause / Initiating Event	Person Affected	Source of Hazard	Sub Mode / Task	Likelyhood per year (From H Category) ESS-0000004	Severity	Likelyhood*Severity	Actions to Mitigate Risk (Risk Controls)
D03	Primary Spectrometer	Bunker	1	Proton beam Off	Access to the bunker during maintenance	Exposed worker without radiation safety task	Activated instrument components. Choppers/Guides/ Monitors	N/A	H1 1	dose < 2 mSv/event		a) Shielding and barriers to prevent access to the bunker area beyond where the work takes place b) Signage.
D03	Primary Spectrometer	Bunker	2	Proton beam Off	Access to the bunker - Barriers and signage	Exposed worker without radiation	Activated instrument components. Choppers/Guides/	N/A	H3 1X10-3	2 < dose < 10 mSv/event		Radiation safety training required by all workers prior to accessing site. Badge access only. MIRACLES staff and users do not have access. Managed by bunker risk assessment.
								N/A	H3 1X10-3	2 < dose < 10 mSv/event		Radiation safety training required by all workers prior to accessing site. Badge access only. MIRACLES staff and users do not have access. Managed by bunker risk assessment.
								N/A	H3 1X10-3	dose > 20 mSv/event		Check of bunker prior to closing. Audio and visual alarm prior to proton beam opening. Emergency stop of proton beam.
								N/A	H4 1X10-6	dose > 20 mSv/event		Check of bunker prior to closing. Audio and visual alarm prior to proton beam opening. Emergency stop of proton beam.
								N/A	H4 1X10-5	dose > 20 mSv/event		Radiation safety training required by all workers prior to accessing site. PSS lock of bunker.
								N/A	H3 1X10-3	dose > 20 mSv/event		Radiation safety training required by all workers prior to accessing site. PSS lock of bunker.
								N/A	H1 1	dose < 2 mSv/event		Guide shielding to achieve radiation levels equal or less than 3 microSv/year on touch. Pit prevents radiation with beam on but this is with



MIRACLES: rescoping and impact in science

A **second analyzer**, another radial collimator and ^{3}He detectors **on the right side**. The upgrade can be done **without any disruption of the operation program**, since all the integration, installation and adjustment features were already manufactured, in anticipation to this completion exercise.

Strategy for rescoping

The feasibility of Si(333) reflections to carry out QENS measurements need to be demonstrated, so the completion needs to be done during hot commissioning.

Thus, the strategy for the rescoping we propose is the following:

1) To install the detector banks of the right side + preamps (the digitizer will be available with extra channels for this right-side ^{3}He tubes).
2) To install the right-side radial collimator.
3) To install the frame of the analyzer, prior to hot commissioning.
4) To install ½ of the right-side analyzer panels with Si(111) crystals (we recommend the top part).

To wait until Si(333) reflections are tested to decide the completion of the bottom part with either Si(111) crystals or Si(311) crystals.

MIRACLES: rescoping and impact in science

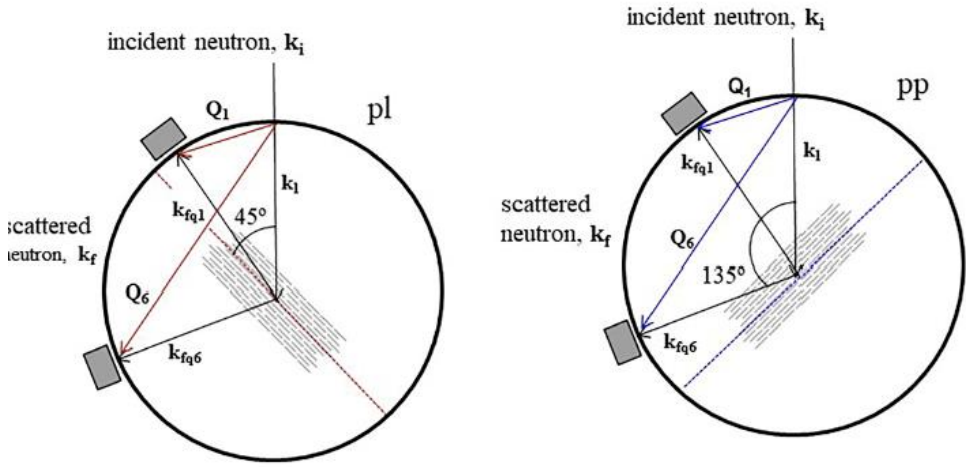
Flux increase (and likely Q-range increase)

- If Si(333) reflections work, neutron flux could double, enabling exploration of new phenomena requiring high flux.
- If Si(333) is not viable, using Si(111) and Si(311) will still increase flux by 50%, expand the Q range, and enhance bandwidth around elastic peaks, potentially revealing confined diffusion effects at the atomic scale.

Anisotropy dynamics

It is interesting to test the anisotropy of the dynamics of water in some materials, like oriented films. An example can be shown in hydration in layered clays.

- Full coverage will allow simultaneous transmission and reflection mode measurements under identical conditions, unlike the current setup, which requires rotating samples for separate measurements.



Applied Clay Science 201 (2021) 105928 & J-Parc new data.

Status of MIRACLES instrument (Oct'24)

Installation (bunker): 2024
Installation (E01): 2024-2026
Installation (E02): 2025
TG5: 2027

Out-of-bunker Guide (ESS-B)
Des. & Mfg. SwissNeutronics
Delivered: Sep'24

Out-of-bunker Guide supports (ESS-B)
Des. & Mfg. ESS bilbao
Delivery: '24-'25

Beamline Shielding (ESS)
NSS Common Project

Thermal shutter (ESS-B)
Des. & Mfg: 23 ESS bilbao
Delivery: Nov'24

Chopper racks (ESS)
NSS Common Project

Choppers (all) (ESS-B)
Des. & Mfg. AERnnova
FAT: Oct
Delivery: Nov '24

Sample Positioning System (ESS-B)
SubTG3: Feb'24
Delivery: Jan '25

Cave & SPAs (ESS-B)
SubTG3: Oct'24
Tender (Mfg.) Dec '24
Delivery: Jan'26

Beam Monitors (ESS)
NSS Common Project

Control Room (ESS-B)
FM Control Hutch?
Installed: Aug'24

BWI (ESS-B)
Des. & Mfg. S-DH
Delivery: Apr'24

Vessel (ESS-B)
Des. & Mfg. AVS
FAT approved
Delivery: Nov '24

NBOA (ESS-B)
FAT Sep '21
Delivery: Oct '21
SwissNeutronics

In-bunker Guide (ESS-B)
Des. & Mfg SwissNeutronics
Installed: Jun'24

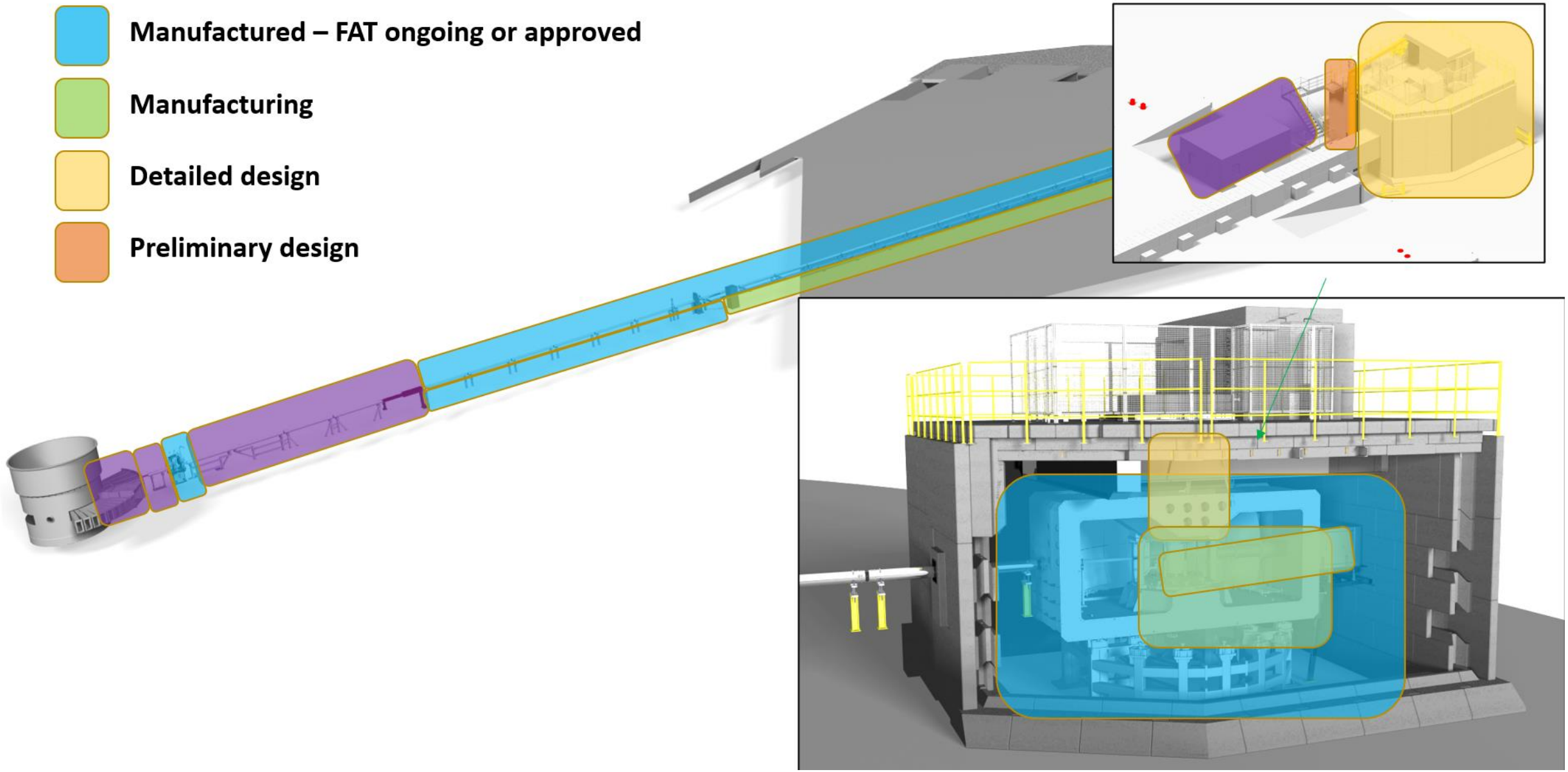
In-vessel (ESS-B)
Des. & Mfg. AVS
Delivery: Jan-Sep '25

In-bunker Guide supports (ESS-B)
Des. & Mfg. ESS bilbao
Installed: Apr'24

Get-lost tube / Beam stop (ESS-B)
Des & Mfg. ESS bilbao
Delivery: Dec '24

Maturity Level (01/10/2024)

- **Installed**
- **Manufactured – FAT ongoing or approved**
- **Manufacturing**
- **Detailed design**
- **Preliminary design**



MIRACLES core team

- Mechanical Engineers (also vacuum and survey/alignment)


Alex Conde


Iván Aranda


Aitor Zugazaga

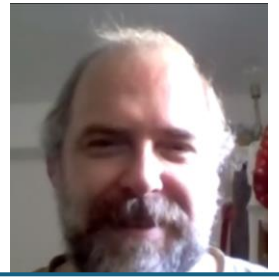
- Control and Electrical Engineers


Idoia Mazkiaran


Giles Harper

- Neutronics and Data Scientists


Octavio G del Moral


Roberto Martínez

- Scientists


Jose E. M. Pereira


Heloisa N. Bordallo


Félix J. Villacorta

Thank you!