Νοτος at ALBA: Versatile XAS and XRD for operando experiments

V ALBA User's meeting

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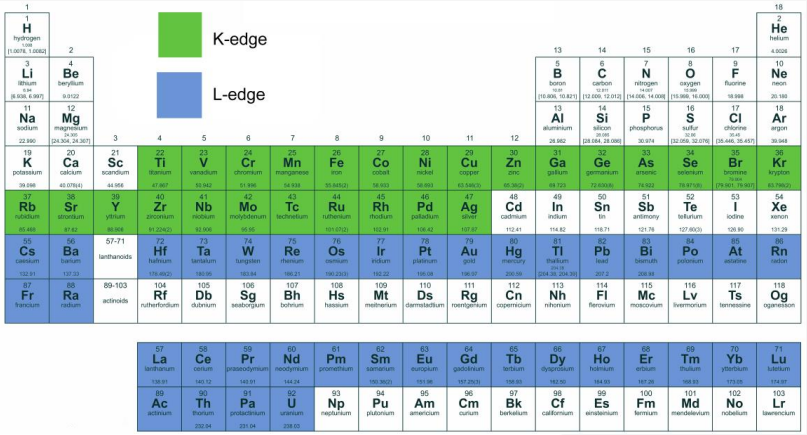
Νότος : description

Scientific case

- Hard X-ray beamline
- Multi-activities capabilities:
  - X-rays Absorption Spectroscopy (XAS)
  - X-Rays Diffraction (XRD)
  - metrology applications
- Study of the electronic structure, short (XAS) and long range order (XRD):
  - chemistry
  - catalysis
  - energy science
  - nanomaterial and condensed matter
  - environmental science
- Infrastructure for *in situ* and *operando* measurements on heterogeneous catalysis and electrochemistry
- Tests and improvement of high precision mechanical components, performances of detector systems, carrying out new methodologies and concepts to improve beamline instruments



Main features

- Energy range **4.7 – 27 keV (XAS) / 30 keV (XRD)**
- Minimum photon flux: **10^{11}(ph/s)**
- Harmonic rejection <**10^{-4}** (Si111)
- Spot properties on sample:
  - Multipurpose station below **100x100 µm^2**
  - PD & XAS station: between 100 µm and 3 mm, in horizontal.
  - Vertically, it should be about 1 mm
  - Vertical collimation of the beam on sample better than 20 µrad FWHM.

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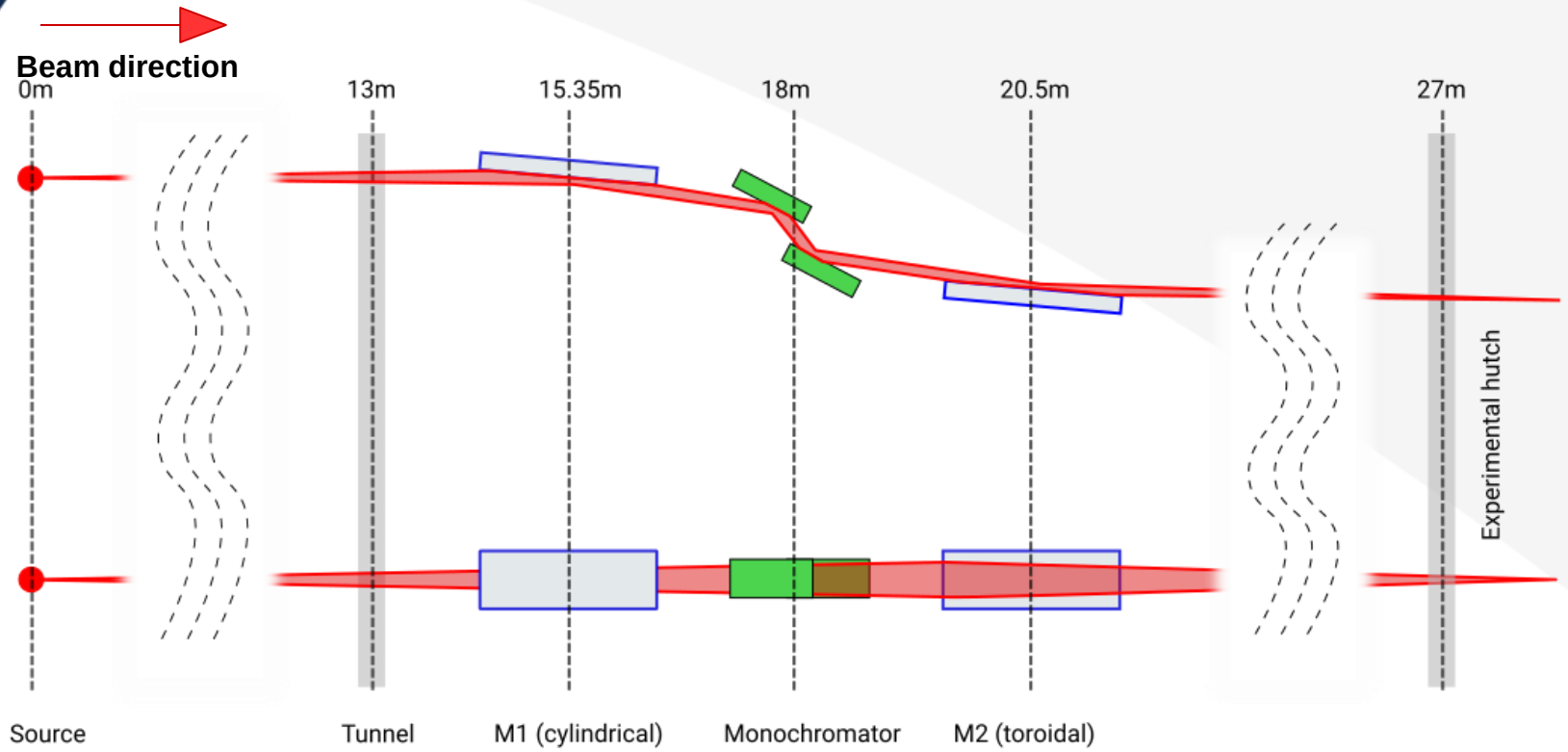
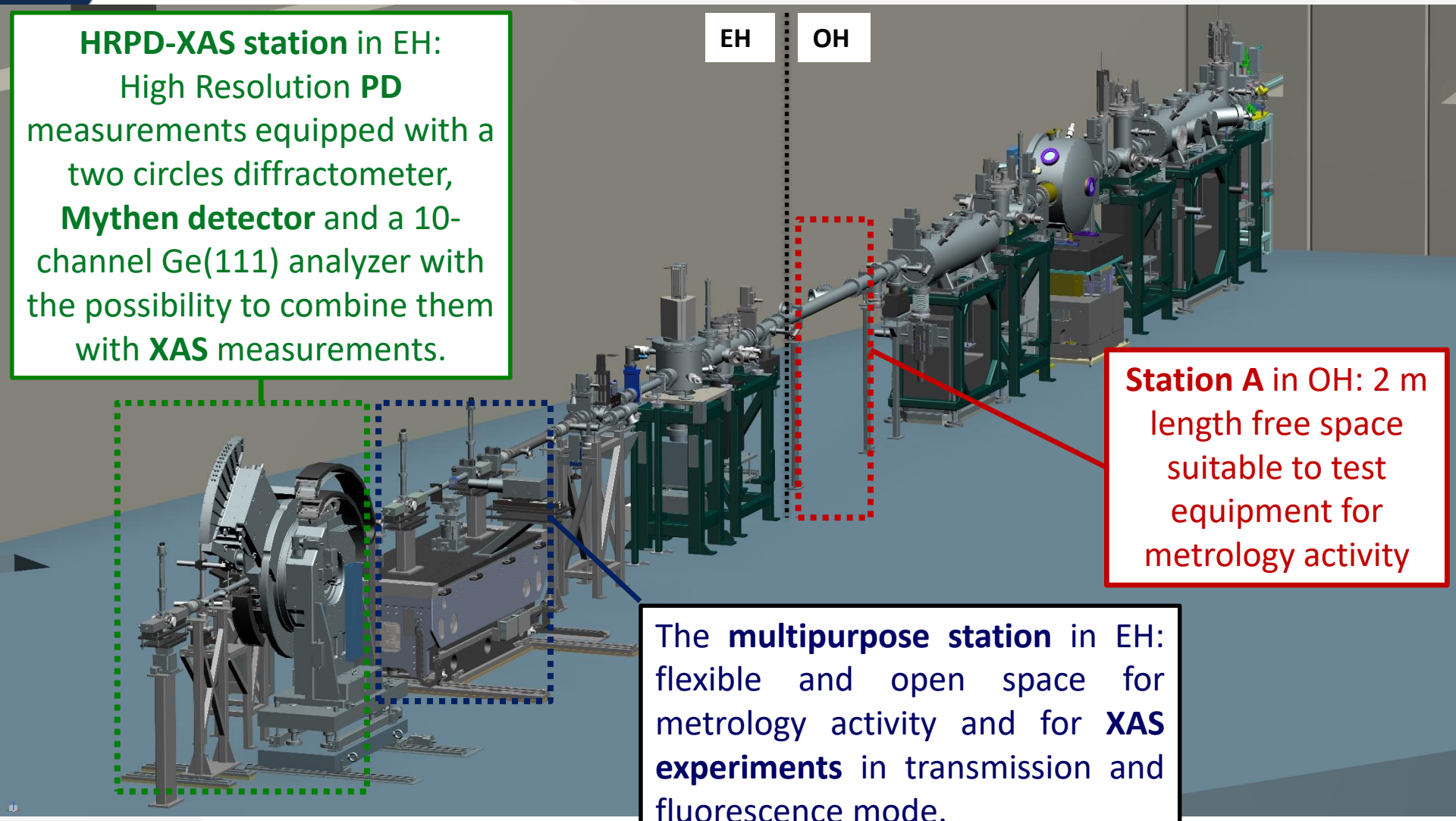
Νότος : Optical layout



- M1 cylindrical dynamic meridional bent mirror for collimation
- Fixed exit Double Crystal Monochromator
- M2 toroidal dynamic meridional bent mirror for focusing

Three Experimental End stations

HRPD-XAS station in EH: High Resolution **PD** measurements equipped with a two circles diffractometer, **Mythen detector** and a 10-channel Ge(111) analyzer with the possibility to combine them with **XAS** measurements.



Station A in OH: 2 m length free space suitable to test equipment for metrology activity

The **multipurpose station** in EH: flexible and open space for metrology activity and for **XAS experiments** in transmission and fluorescence mode.

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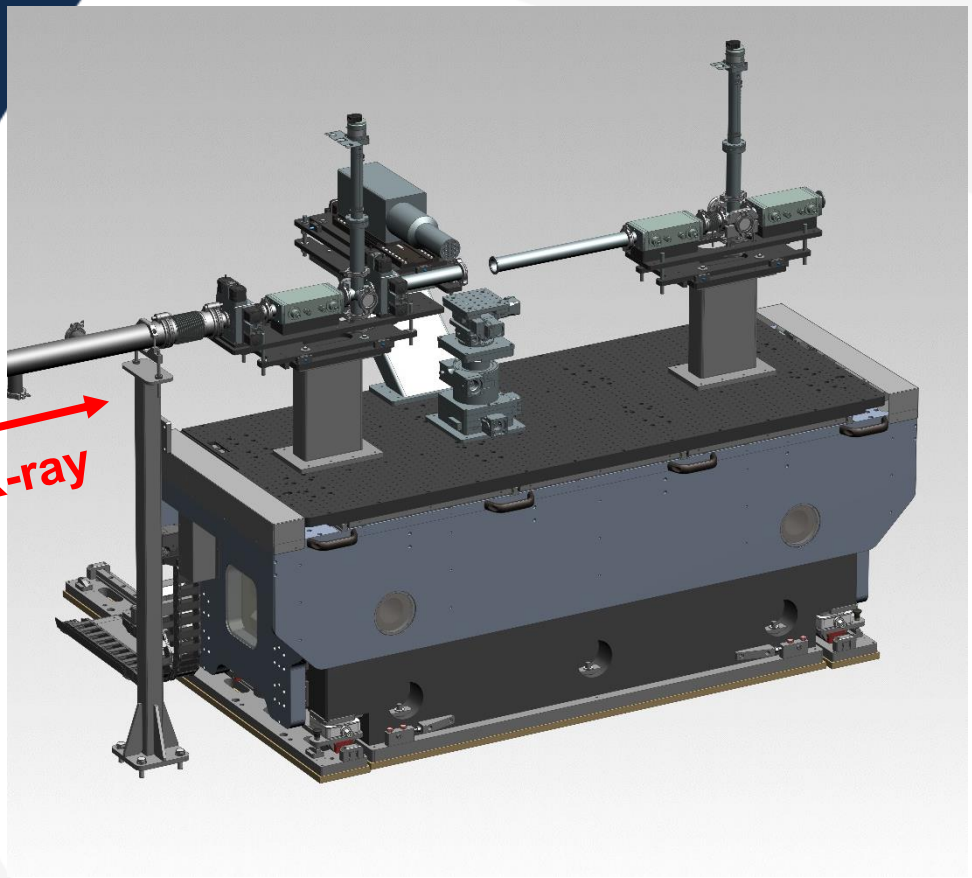
Multipurpose station



Requirements of the station:

- **Flexible** and **open space for metrology** activity and for XAS experiments
- **Open to future developments** in function of needed identified together the users community

Standard configuration for XAS:

- 3 ion chambers for XAS measurements in transmission
- 13-channels SDD 90° with respect X-Rays beam
- Motorized stage at sample position
- 2 sample stage for standard and references

Installation on going

X-Ray commissioning in Autumn 2022

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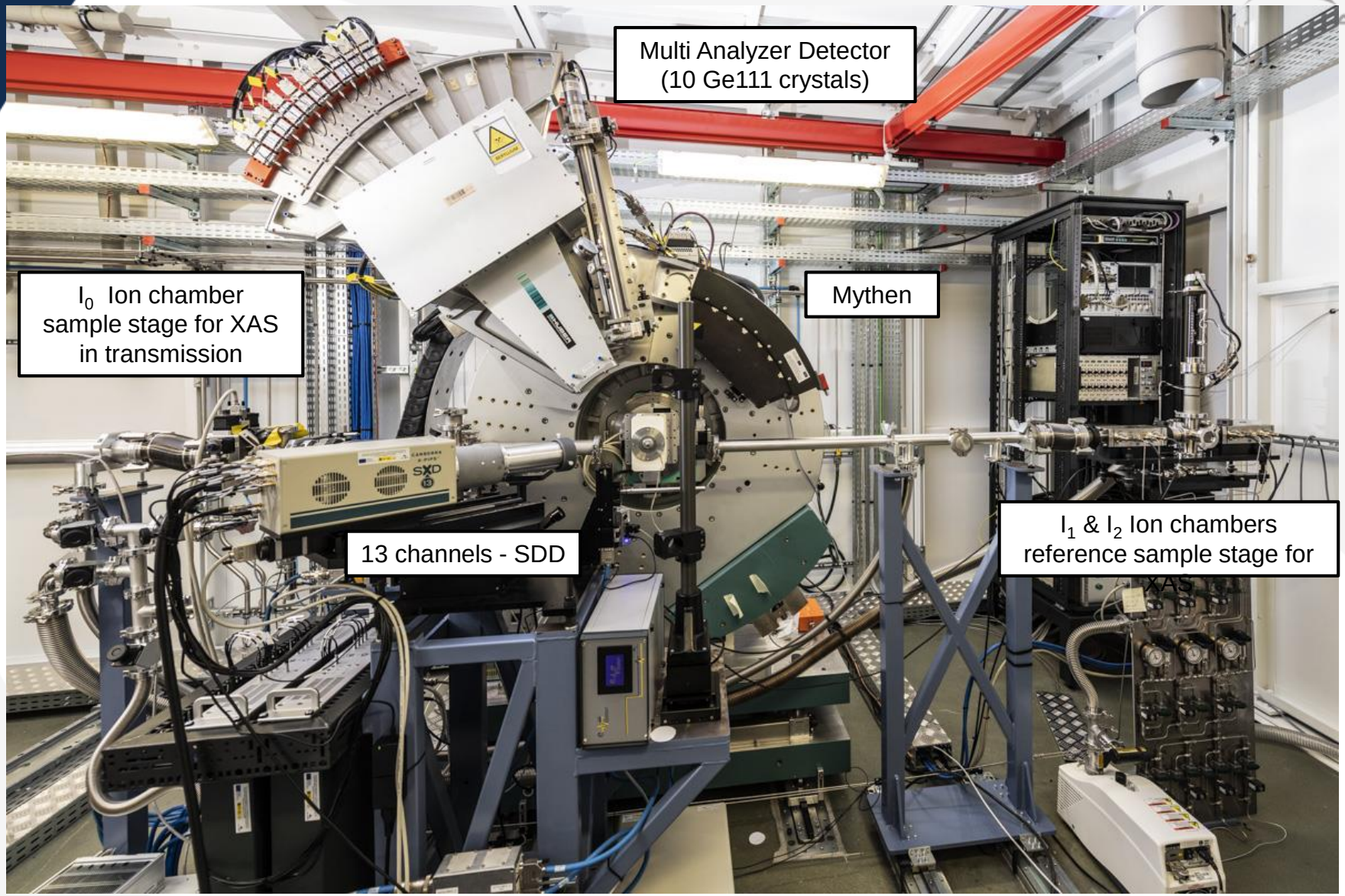
EH: HRPD&XAS station



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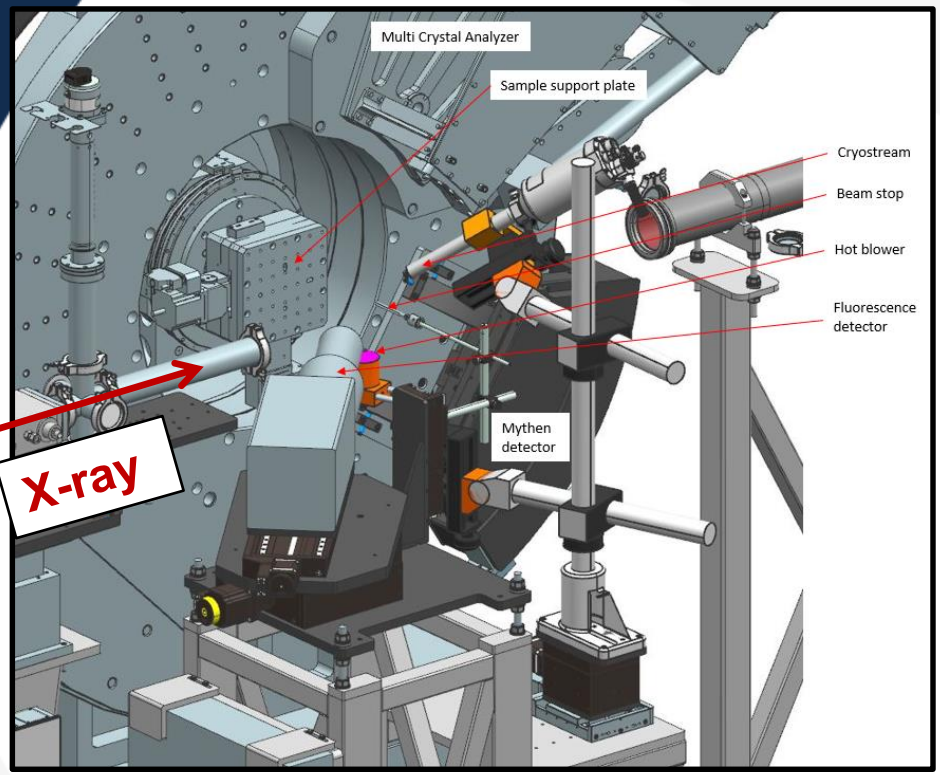
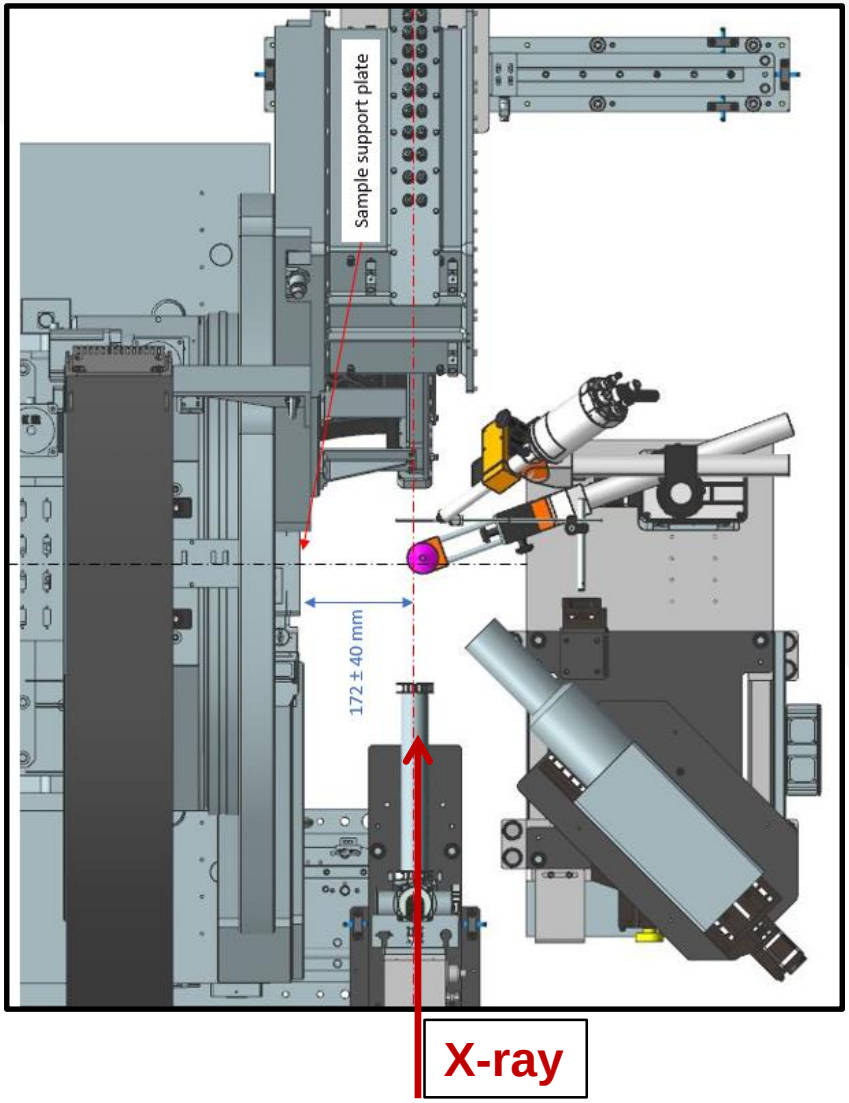
HRPD&XAS station: space at sample position





Possibility to mount **different sample environments** at sample position but with some space constrains

More information document session of the website:
https://www.albasynchrotron.es/en/beamlines/bl16-notos/documents-photos

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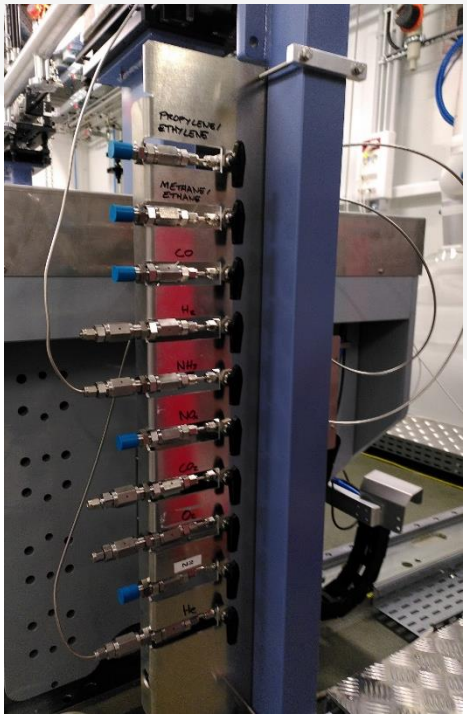
System for reactive gas: Infrastructure

Gas cabinet and gas cylinder



Supply point in EH

Extraction in EH

Control panel extraction and alarm system

- High pressure side installation concluded
- Still pending to finalize installation of safety system (detectors + alarm system): planned for Autumn 2022.

Infrastructure suitable to work up to 80 bar

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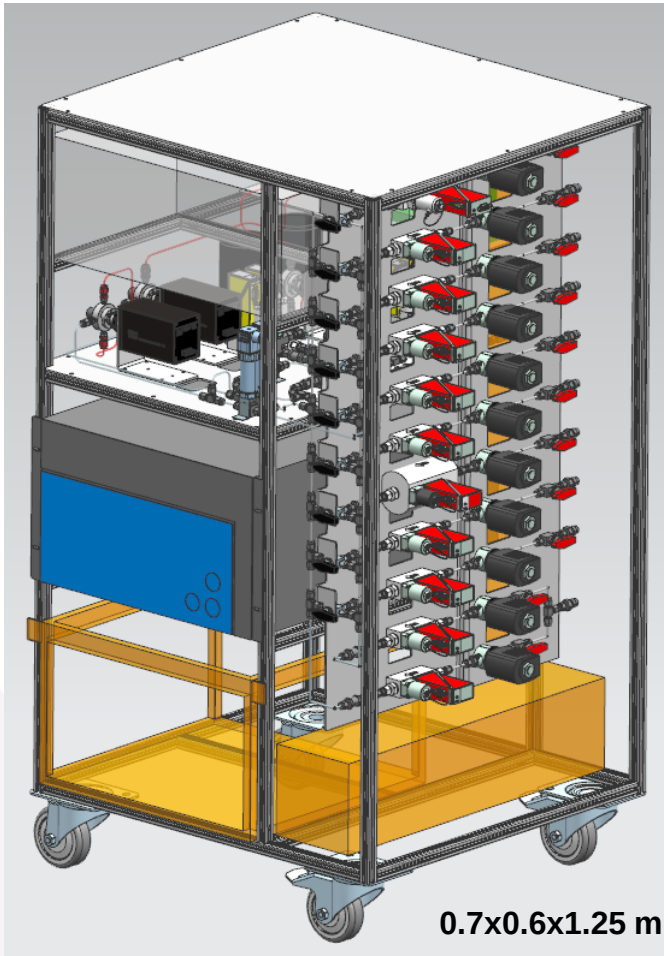
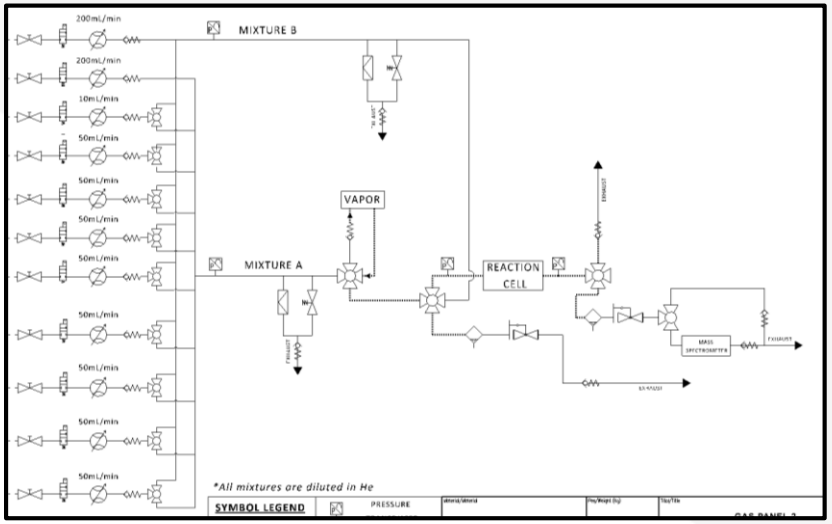
System for reactive gas: delivery to sample environment

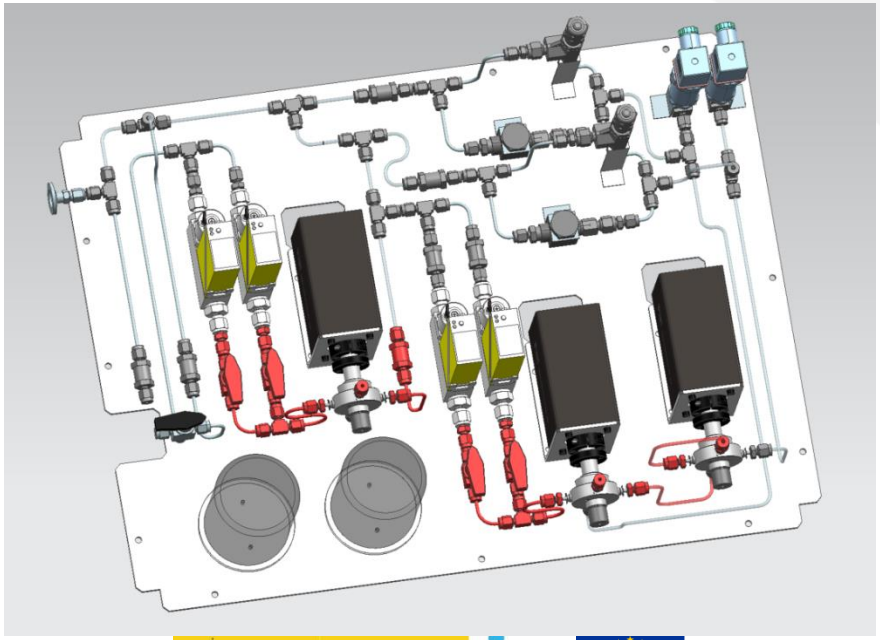
- **High flow system** (MFC 50 ml/min each) in house development

Setup integrated in control system of beamline

Movable trolley to supply gases to sample environment







Melika El Manchoul, Jordi Prat and Edmundo Fraga

Max pressure 24 bar

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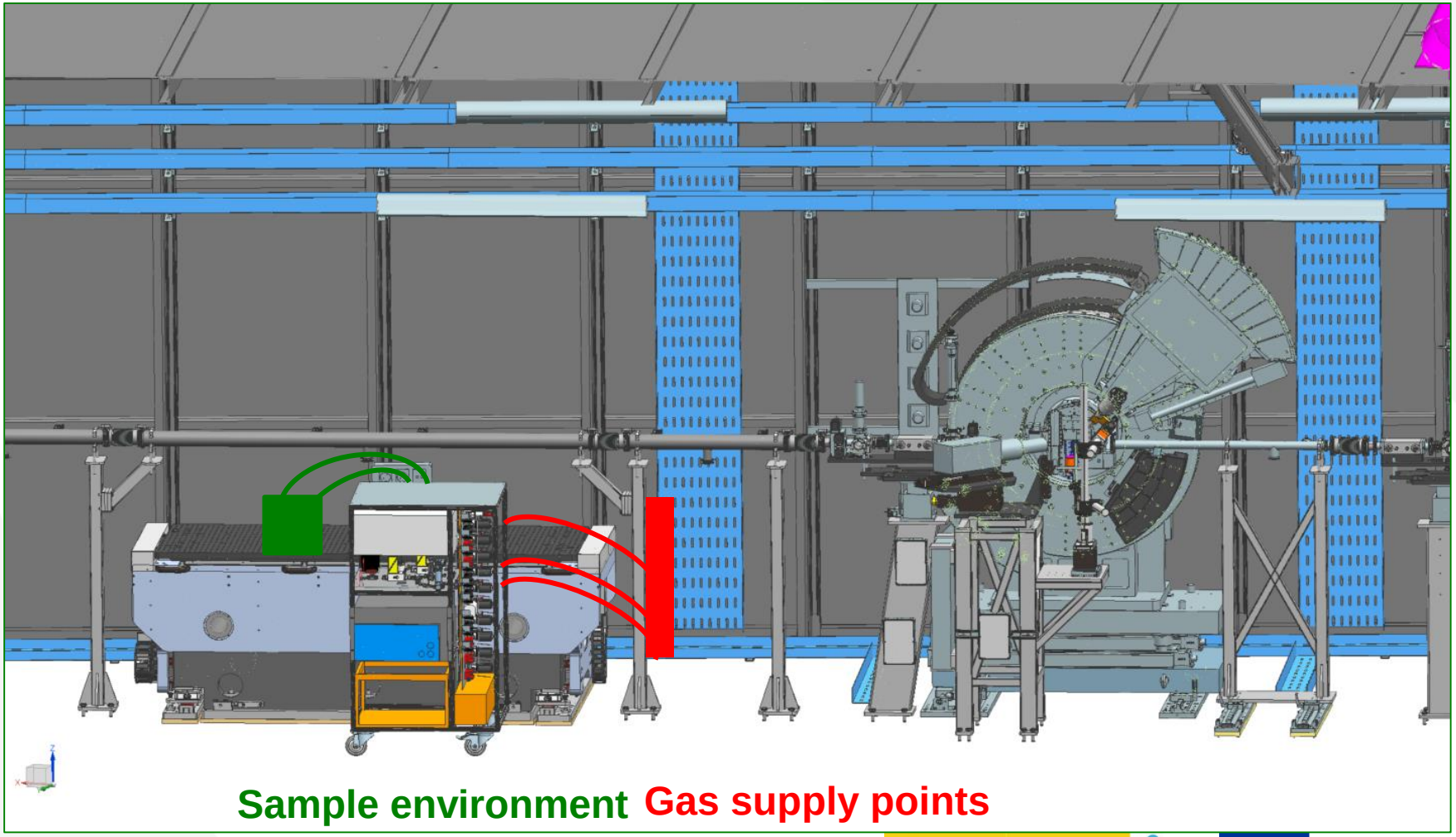
System for reactive gas: delivery to sample environment

- **High flow system** (MFC 50 ml/min each) in house development

ALBA

Beamline integration: multipurpose station



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System for reactive gas: delivery to sample environment

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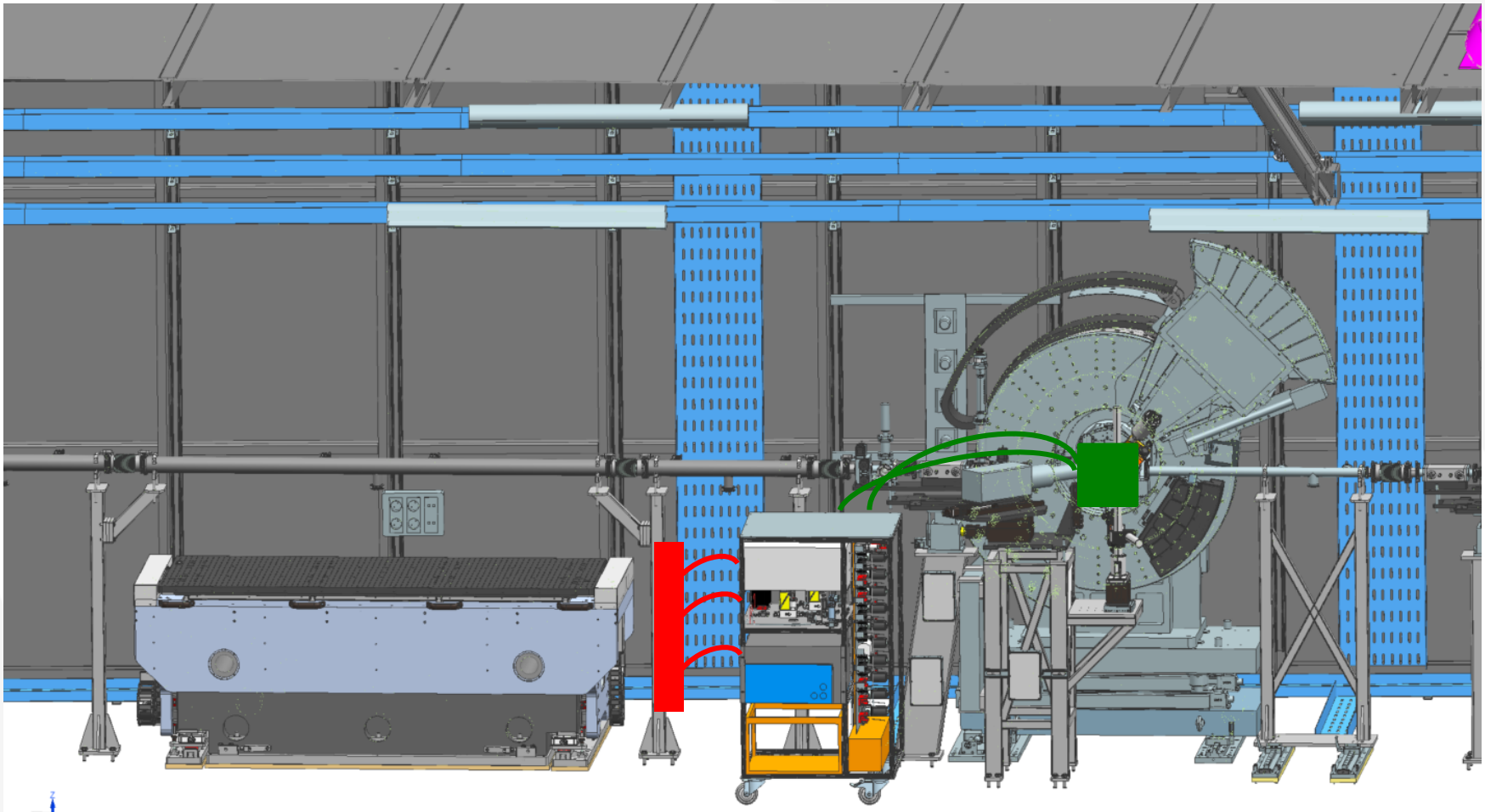
ALBA

Beamline integration: PD&XAS station



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System for reactive gas: delivery to sample environment

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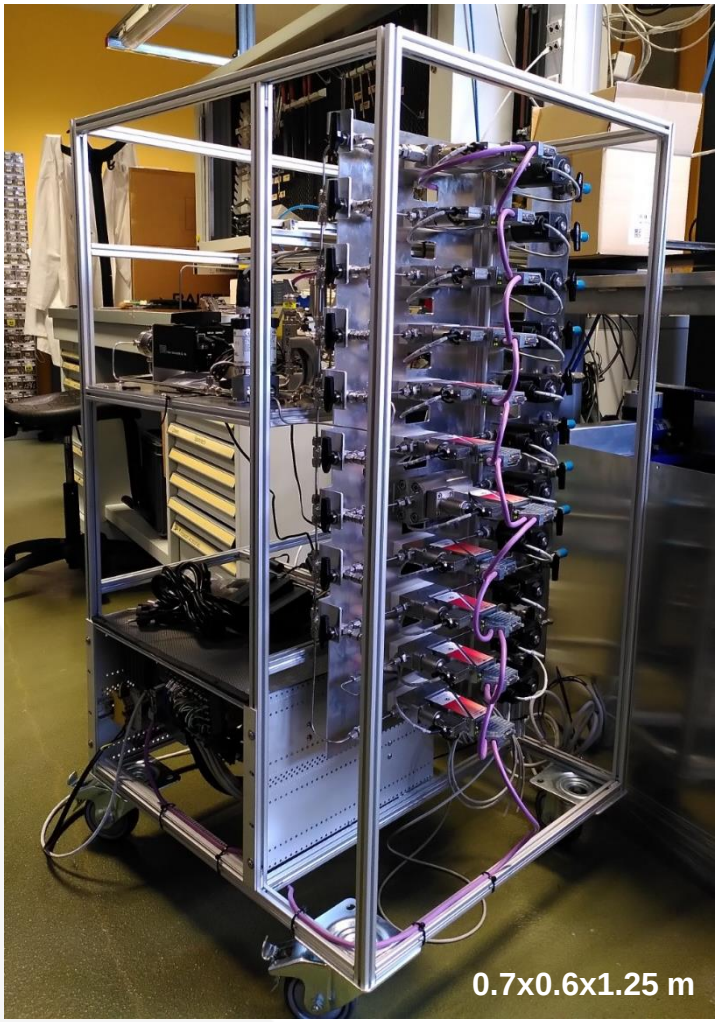
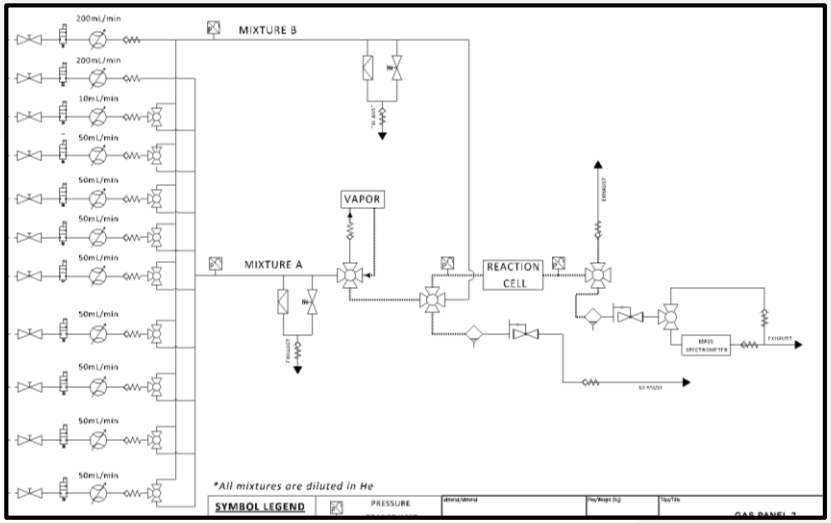
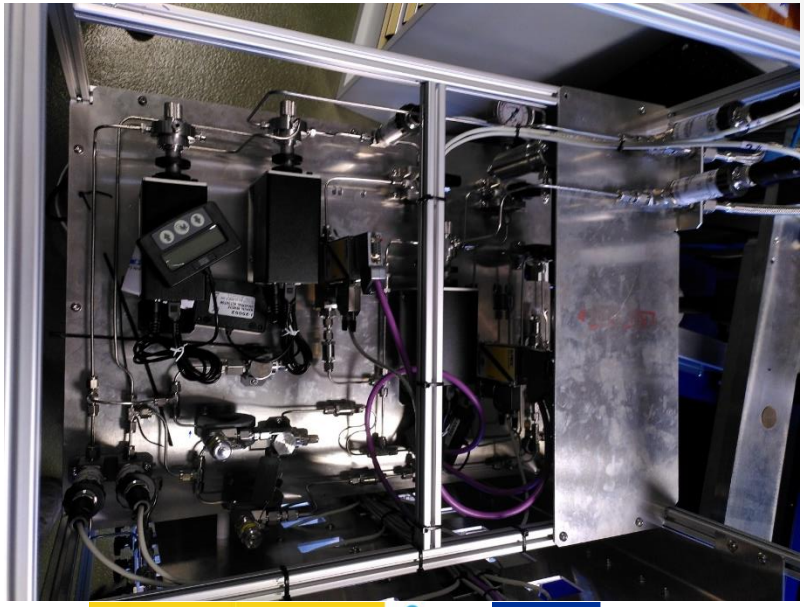
Setup integrated in control system of beamline

Movable trolley to supply gases to sample environment







Melika El Manchoul, Jordi Prat and Edmundo Fraga

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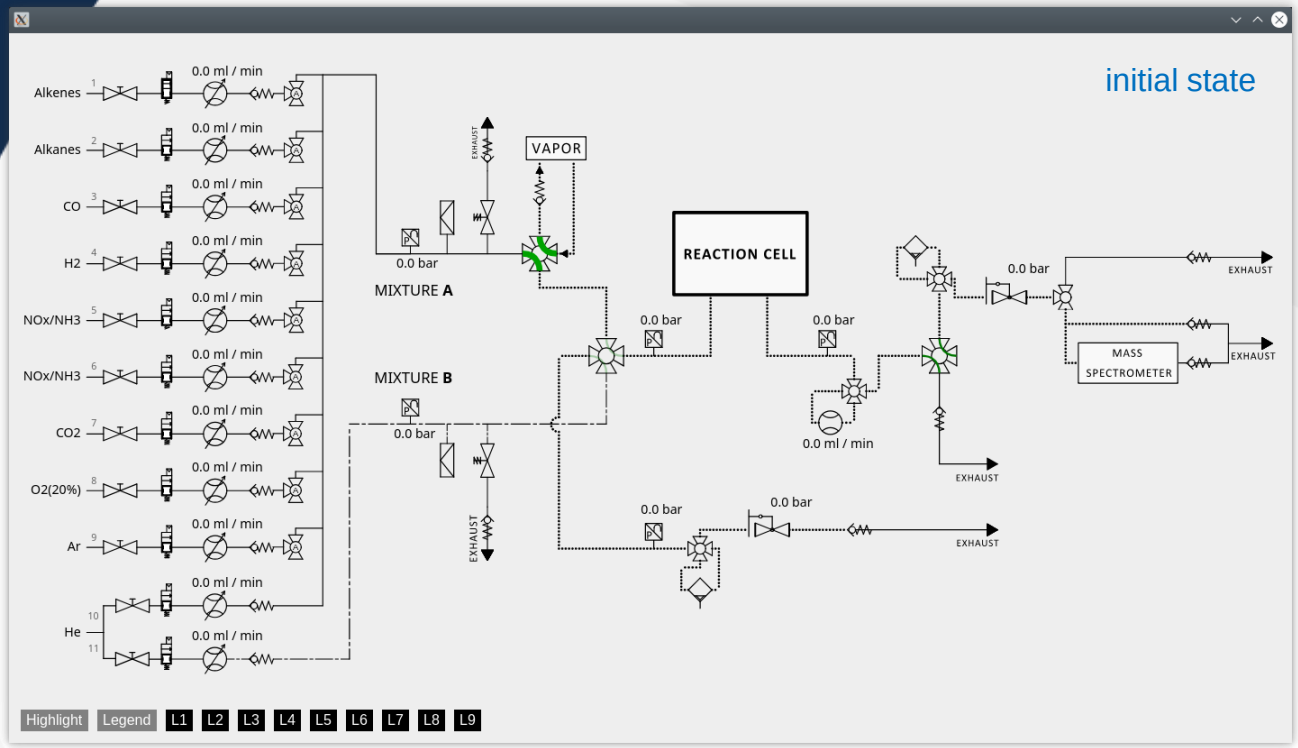
System for reactive gas: delivery to sample environment

- **Graphical User Interface**



The whole setup will be **integrated** in the **control system** of the beamline

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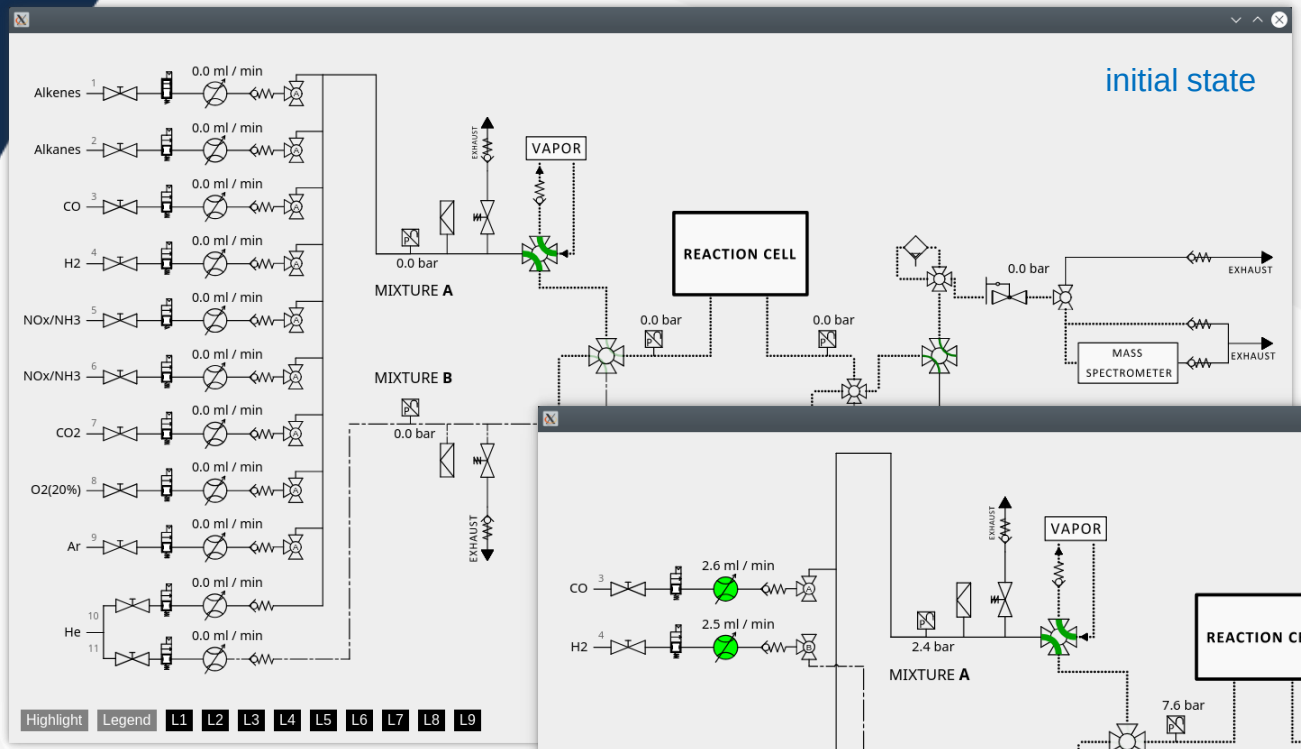
System for reactive gas: delivery to sample environment

- **Graphical User Interface**



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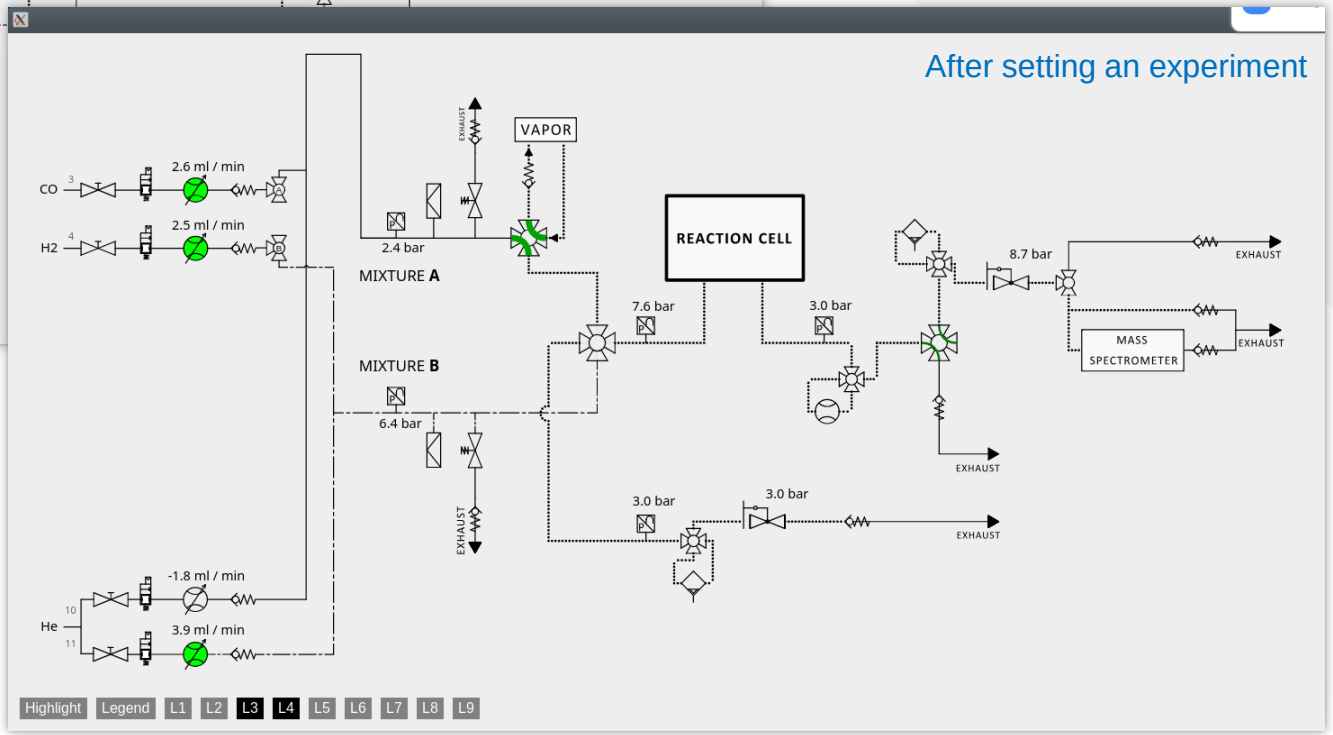

Possibility to configure the graphical interface in function of the experiment

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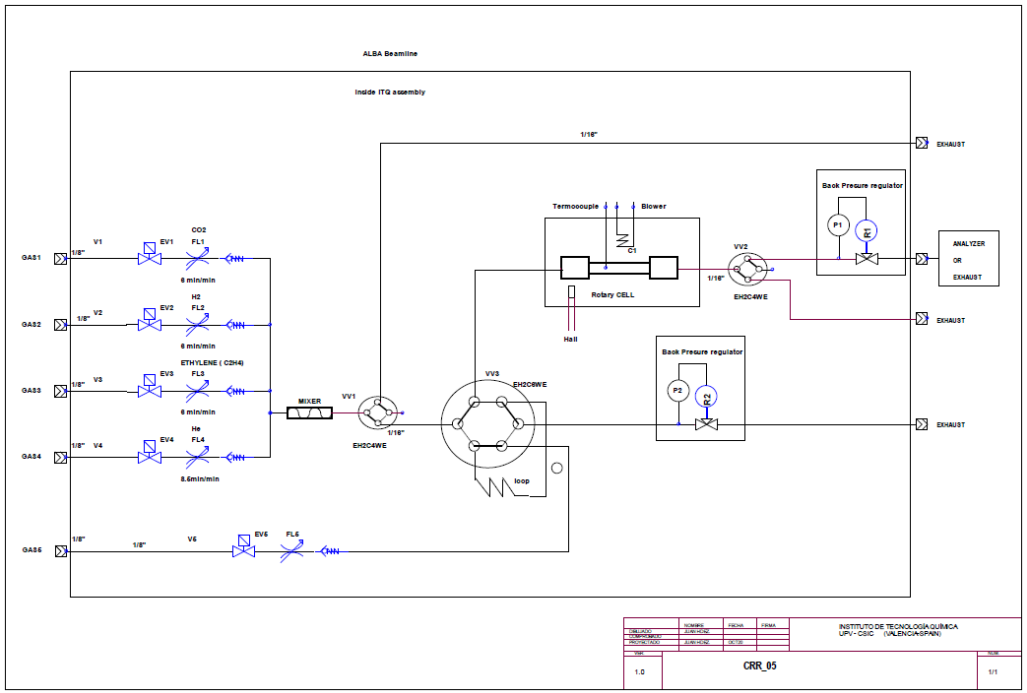
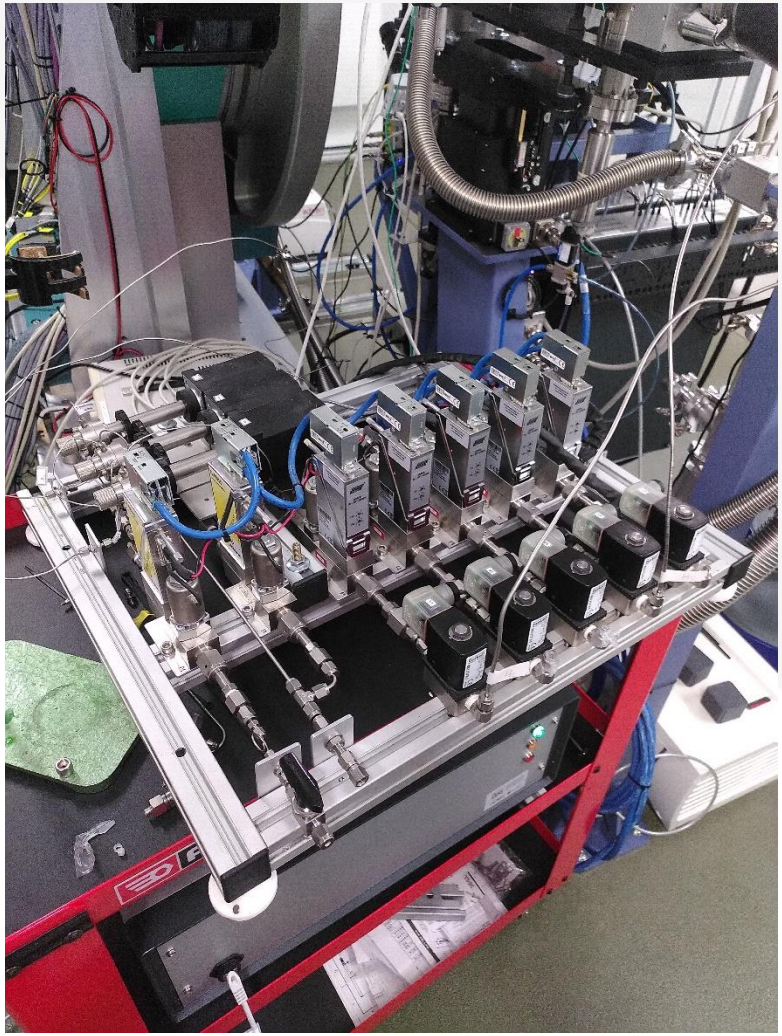
System for reactive gas: delivery to sample environment

- **Low flow system** (MFC 6 ml/min each) – collaboration Alba-ITQ (Valencia)





- **Dead volume ≈17 ml**
- 6-way valve with calibrated loop to send pulses of reactants

This equipment has been developed as part of the RTI2018-096399-A-I00 R&D project, funded by MCIN/AEI/10.13039/501100011033/ and “ERDF A way of making Europe”.

More details by Wilson Henao talk #63 AUSE 06/09/22

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System for reactive gas: Mass Spectrometer

- **MKS Cirrus™ 3-XD**

- **It could be coupled with both gas systems and users' setup** (expected at Alba in Autumn 2022)
- **Integrated** in data acquisition and archiving system of the beamline

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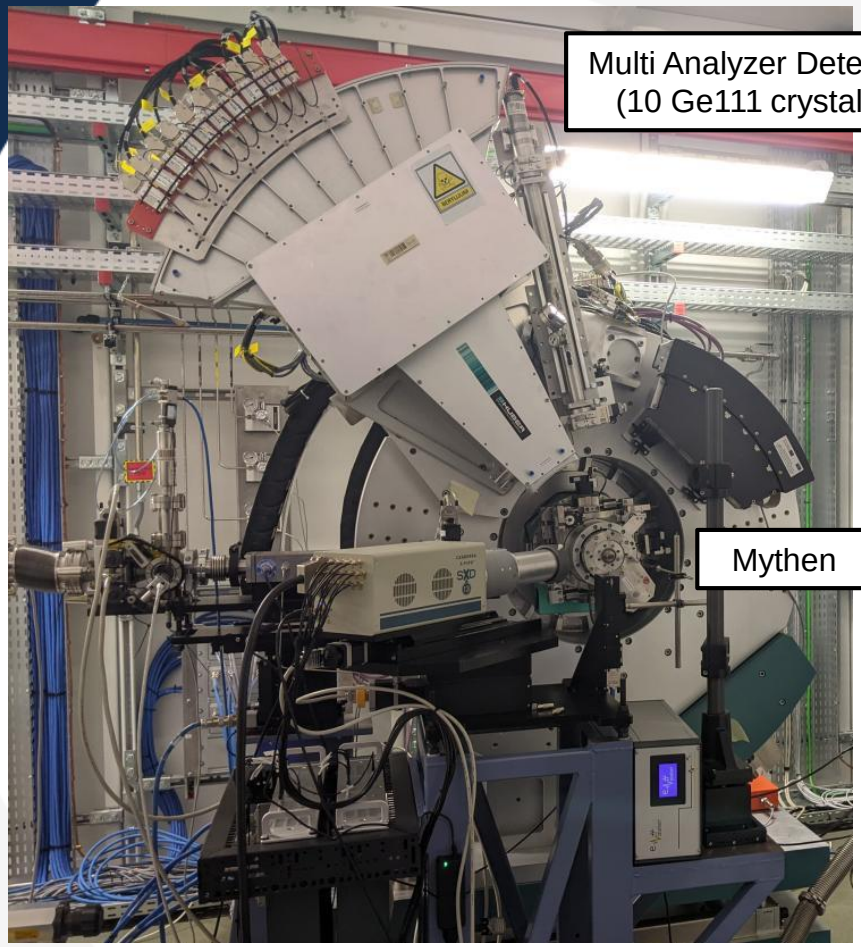
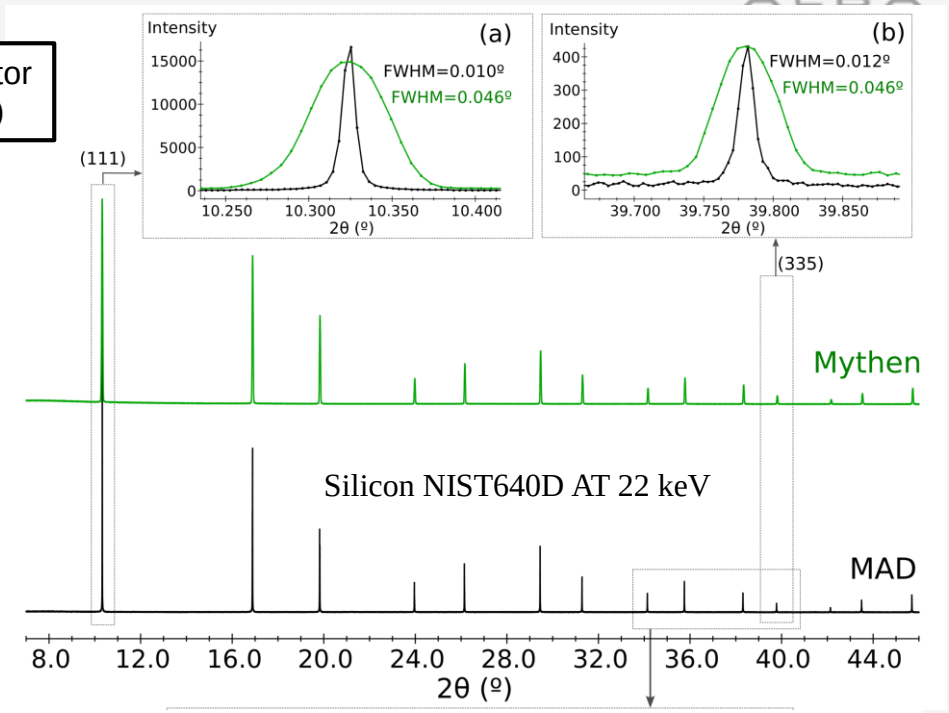
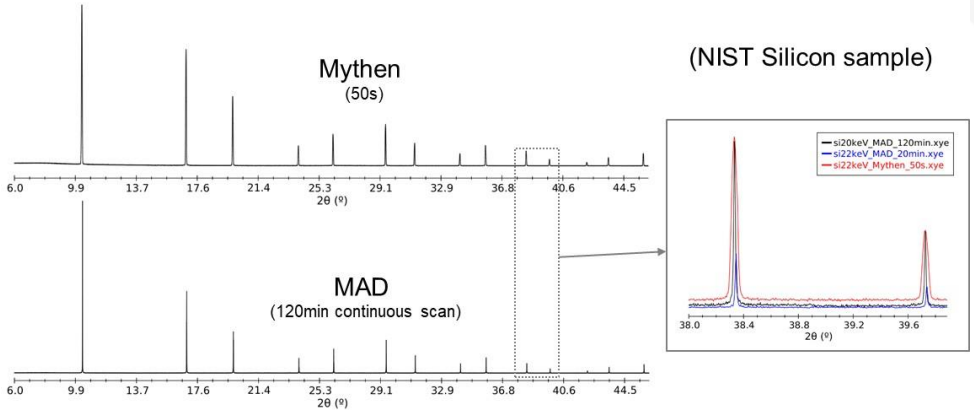
X-Ray commissioning: XRD



Multi Analyzer Detector
(10 Ge111 crystals)

Mythen





- MAD has the highest angular resolution
- Mythen allows higher time resolution

Measurement performed without M2

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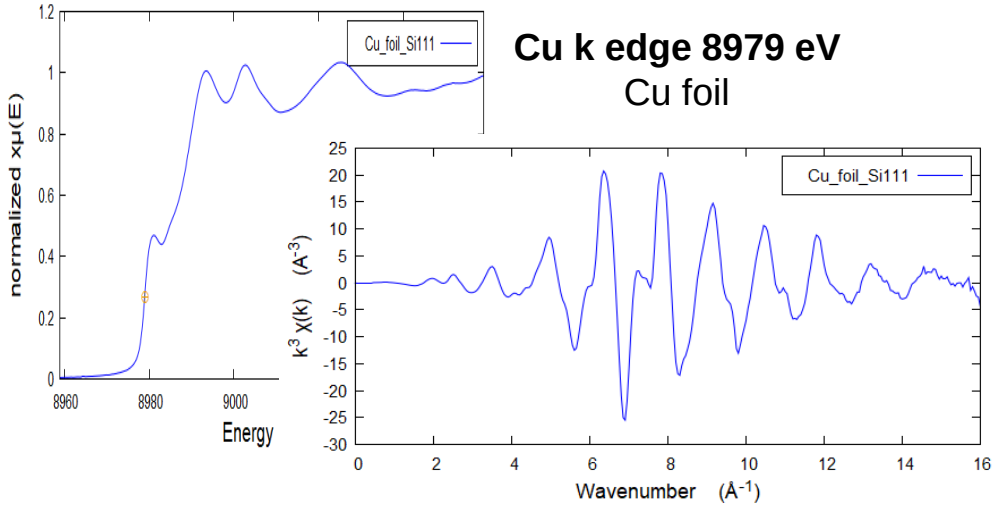
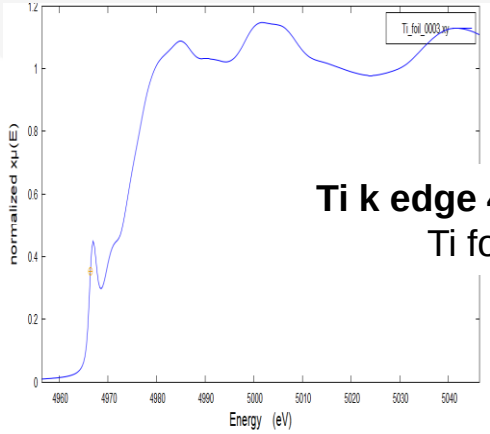
X-Ray commissioning : XAS

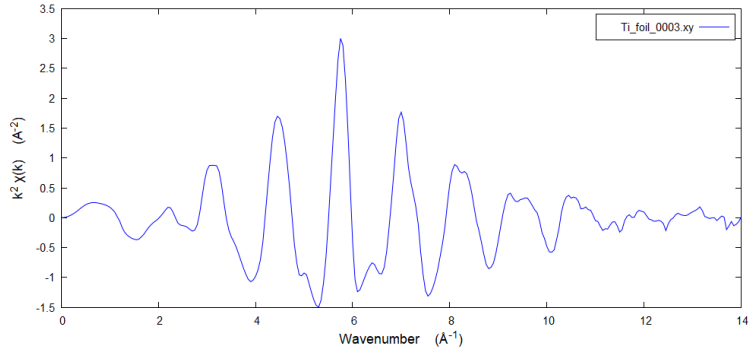
- XAS measurements in continuous mode
- 13-channels SDD available to the users (0.5 mm carbon as temporary window)

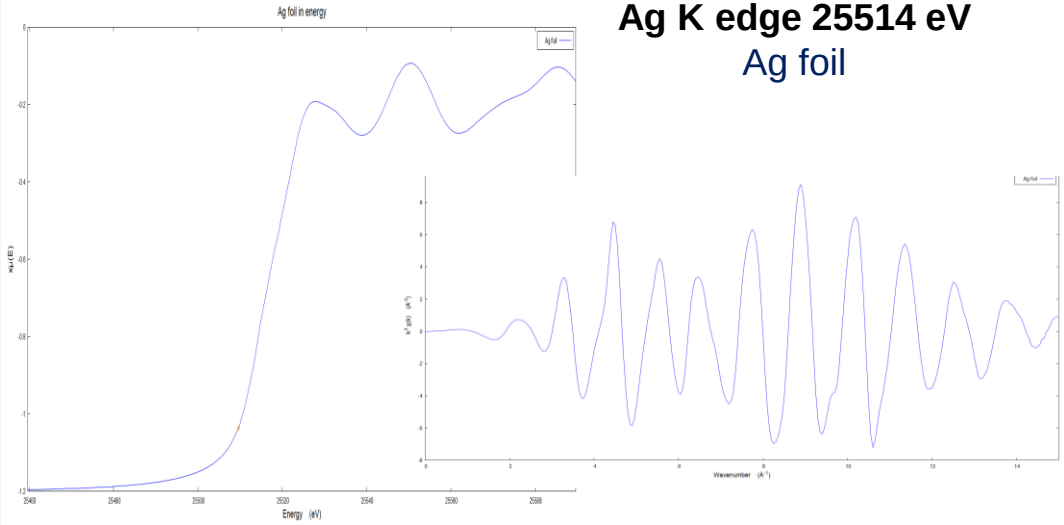


Cu k edge 8979 eV
Cu foil



Ti k edge 4966 eV
Ti foil





Ag K edge 25514 eV
Ag foil

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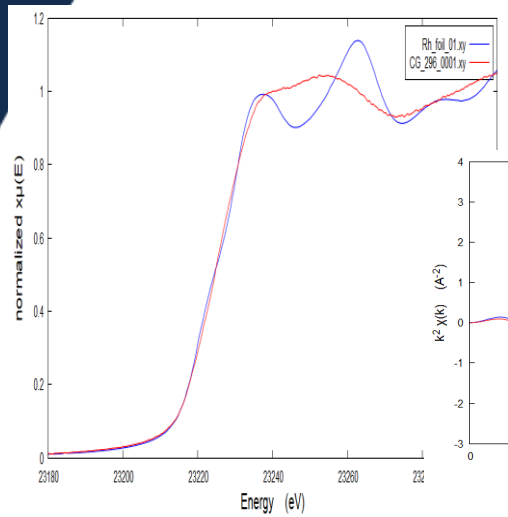
X-Ray commissioning : XAS

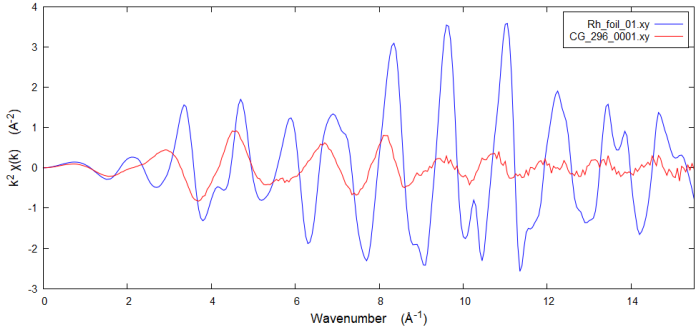
- Rh coating on the mirrors

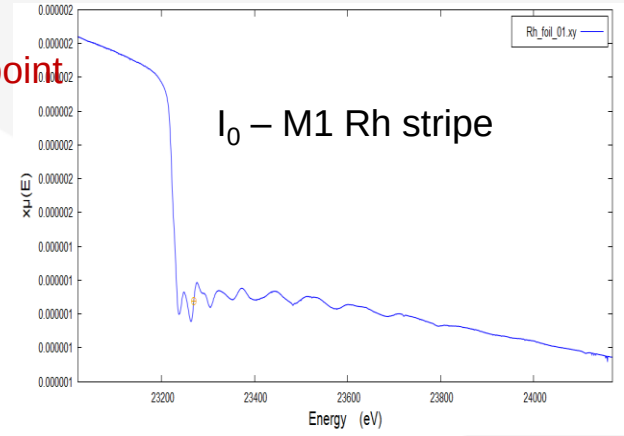


Rh k edge 23220 eV

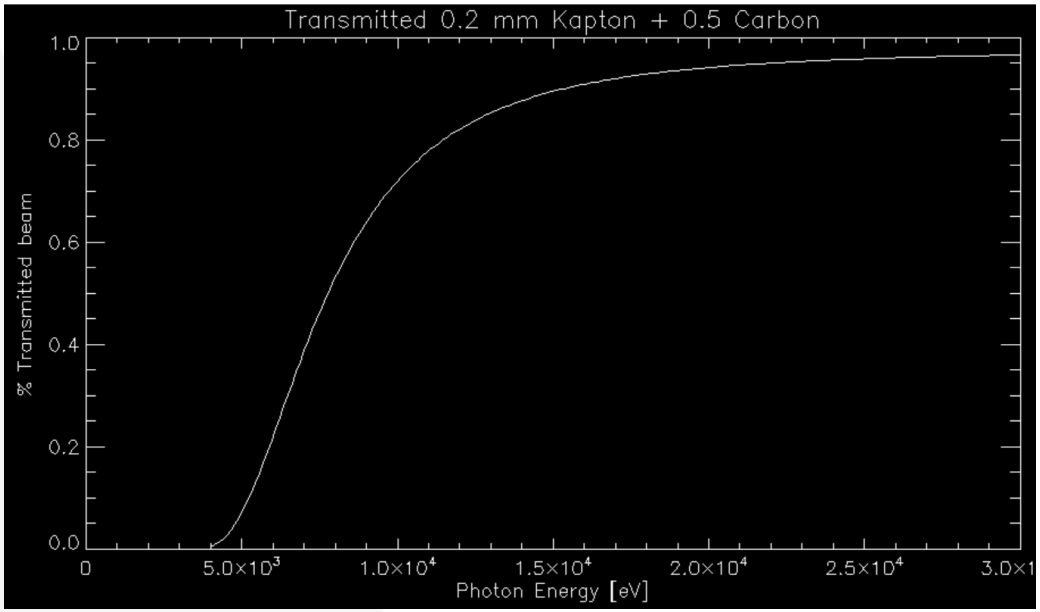
Rh foil in transmission

Catalyst 1%Rh/C in fluorescence T=0.2 s/point





200 μm Kapton window between OH-EH + 13-channels SDD available with 0.5 mm carbon window



Limited photons at low energy in fluorescence configuration: 20 % of beam at 5.9 keV

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Beamtime to users: April – July 2022

Users experiments since end of April: 9 experiments - 99 shifts

- **Rosa Palacin** (CSIC- Barcelona) 26th of April to 1st of May - 15 shifts
  - Combination of XAS and XRD techniques for operando electrochemistry experiment
- **Gonzalo Prieto** (ITQ - Valencia) – 24th -29th of May - 18 shifts
  - Towards the understanding of surface site requirements in metal atom trapping phenomena for the synthesis of single-atom catalysts
- **Wilson Heinao** (ITQ - Valencia) – 31st of May 2nd of June - 9 shifts
  - EXAFS study of local structure of low-atomicity Pt catalysts
- **Nuria Divins** (UPC - Barcelona) – 3rd -5th of June - 9 shifts
  - In situ XAS and XRD measurements of redox cycles of heterogeneous catalysts
- **Oriol Vallcorba** (Alba) – 9th -10th of June - 6 shifts
  - Understanding structural flexibility in crystalline coordination compounds. Two examples of contrasting nature, a flexible Metal Organic Framework and a Porous Molecular Material
- **Ana Iglesias** (CSIC - Madrid) 14th – 16th of June - 9 shifts
  - Investigation by XAS and XRD of Copper/Zinc catalysts for CO_2 hydrogenation
- **Marianne van der Merwe** ((HZB – Berlin) 7th – 10th of July - 12 shifts
  - Correlating potential- and pH induced transformations of highly active iridium-based electrocatalysts with catalytical stability using operando XANES and EXAFS
- **Vera Truttmann** (TUE- Wien)12th – 15th of July - 12 shifts
  - Insights into the structure dynamics of thiolate and phosphine protected gold nanoclusters supported on CeO2 in CO oxidation related to ligand and structure configuration
- **Marine Reynaud** (CIC energiGUNE - Miñano) 19th - 21st of July - 9 shifts
  - Combined XRD-XAS Operando electrochemical experiment at the NOTOS HRPD-XAS end station

Capability to the users for call 2023-I:

Full energy range available

Sample environment: capillary setup and cell for self supported pellet (only XAS in transmission)

Reactive Gas at ambient pressure

Temperature: 80-400 K (cryostream) and RT-900 C (hot gun)

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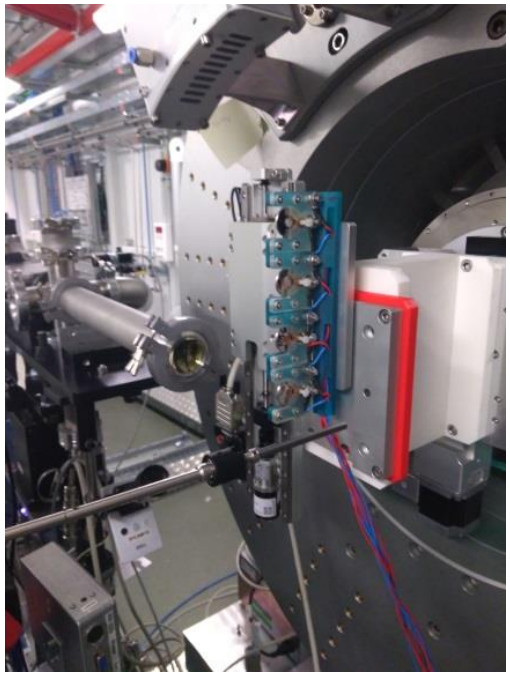
First Expert users: *operando* experiment in battery

Combination of XAS and XRD techniques for operando electrochemistry experiment

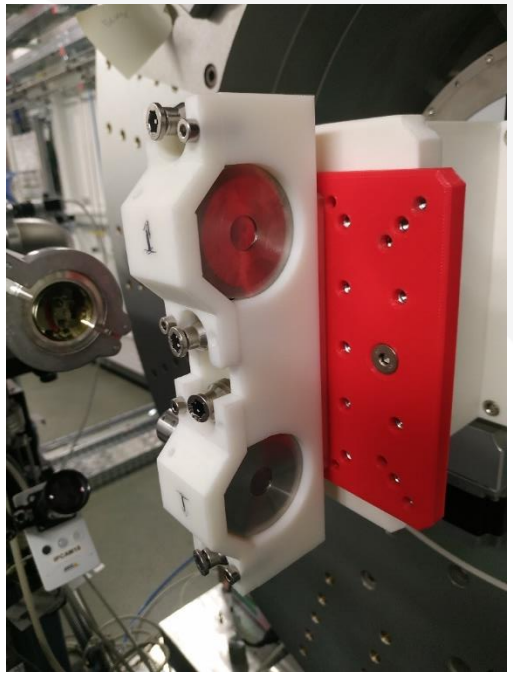
M. Rosa Palacin and Ashley Black - CSIC- Institut de Ciència de Materials de Barcelona

- Commercial (NEI corporation) casted electrodes: NMC and LFP
- XAS in transmission geometry
- PD in transmission geometry by Mythen detector at 11keV

26th of April to 1st of May 2022 at NOTOS



Multistage coin cell



Multistage Leriche cell

More details by Ashley Black talk #69 AUSE 06/09/22

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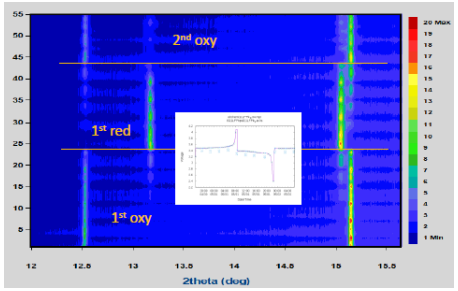
First Expert users: *operando* experiment in battery

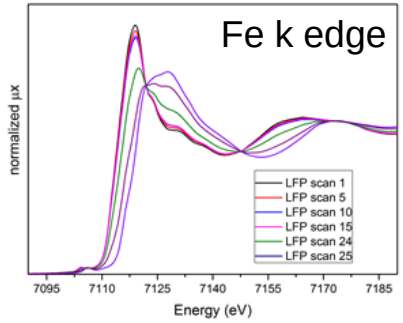
LFP coin cell C/10

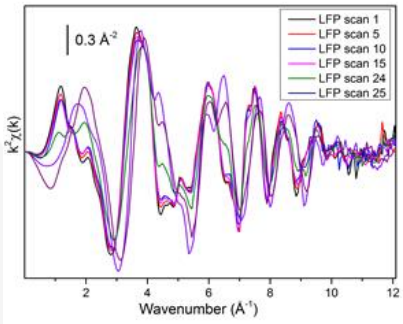
PD at 11 keV

Fe K edge (7112 eV) in transmission geometry

1 measurement cycle ≈ 5’







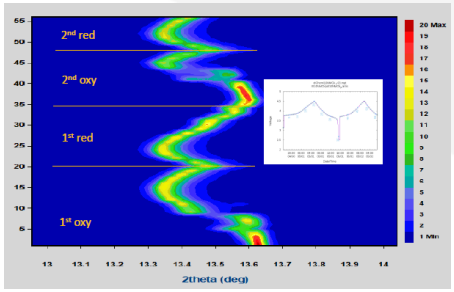
NMC coin cell C/10

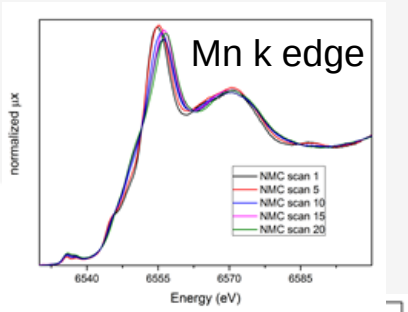
PD at 11 keV

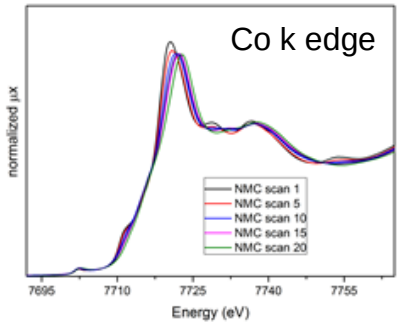
Mn (6539 eV), Co (7709 eV), Ni (8333 eV) k edges

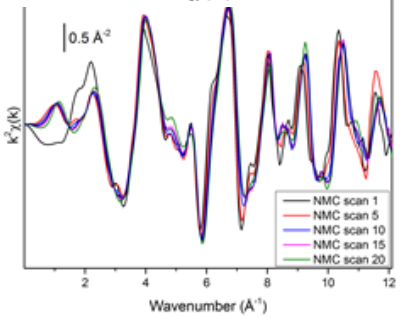
XAS in transmission geometry

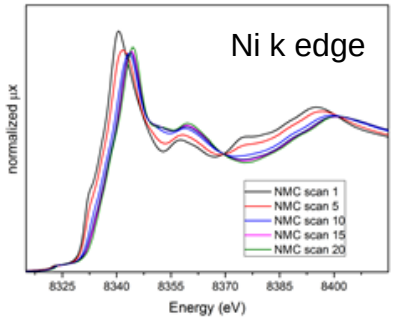
1 measurements cycle ≈11 ‘

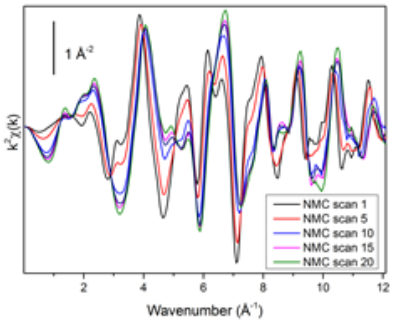












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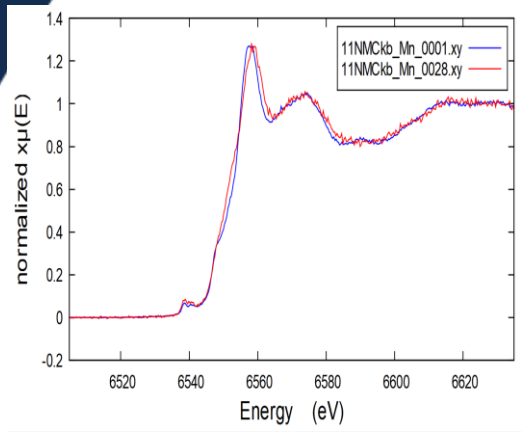
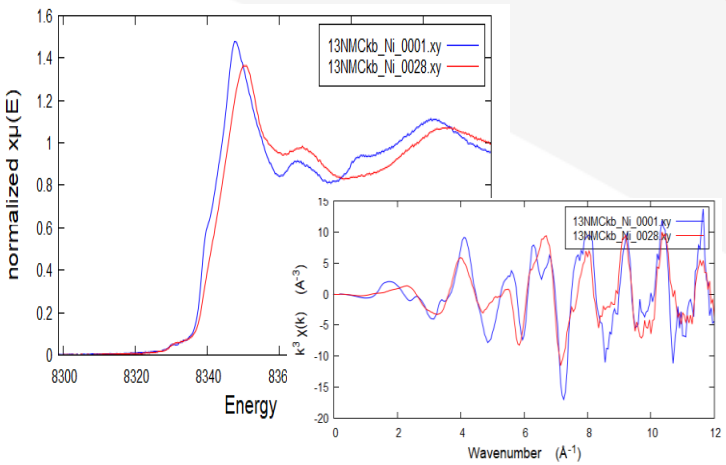
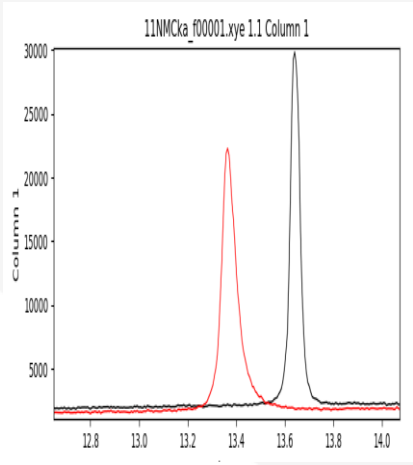
operando experiment in battery

NMC system, Integration time = 0.1 s/point

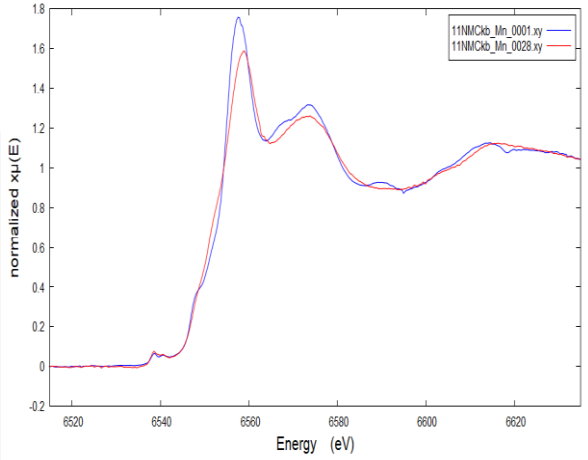
Measurements in fluorescence configuration

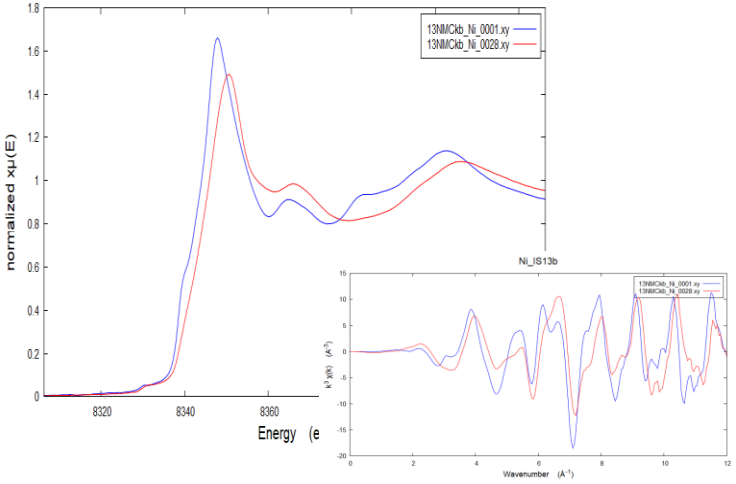


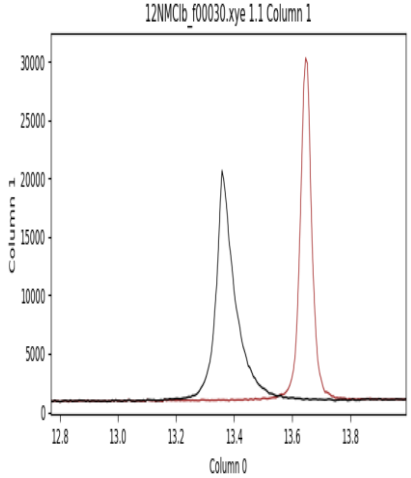




Measurements in transmission configuration







Marine Reynaud (CIC energiGUNE - Miñano) & M. Rosa Palacin and Ashley Black - CSIC- Institut de Ciència de Materials de Barcelona

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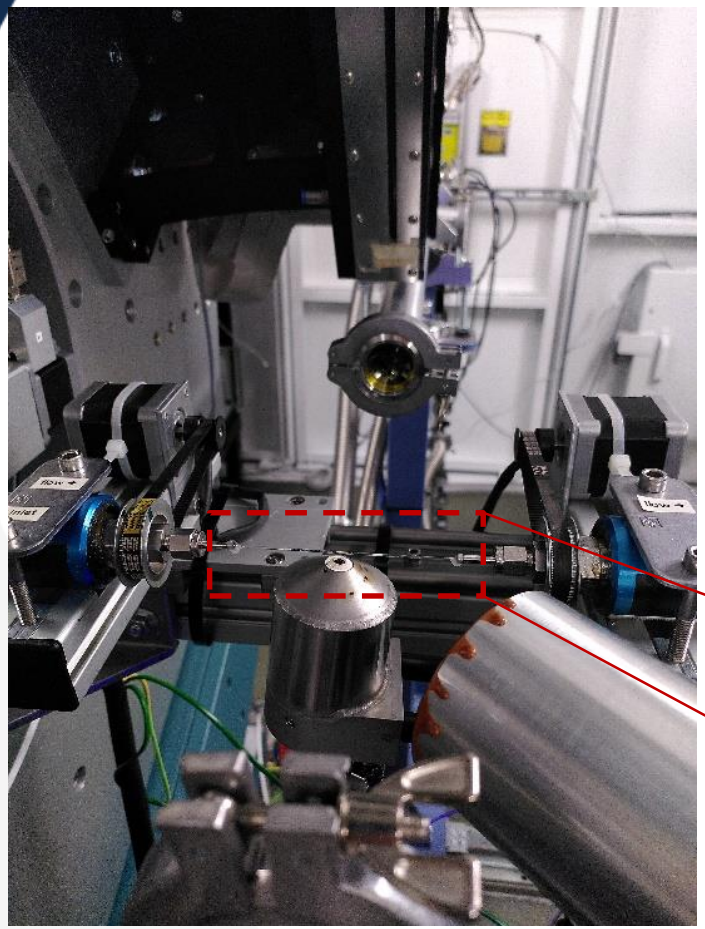
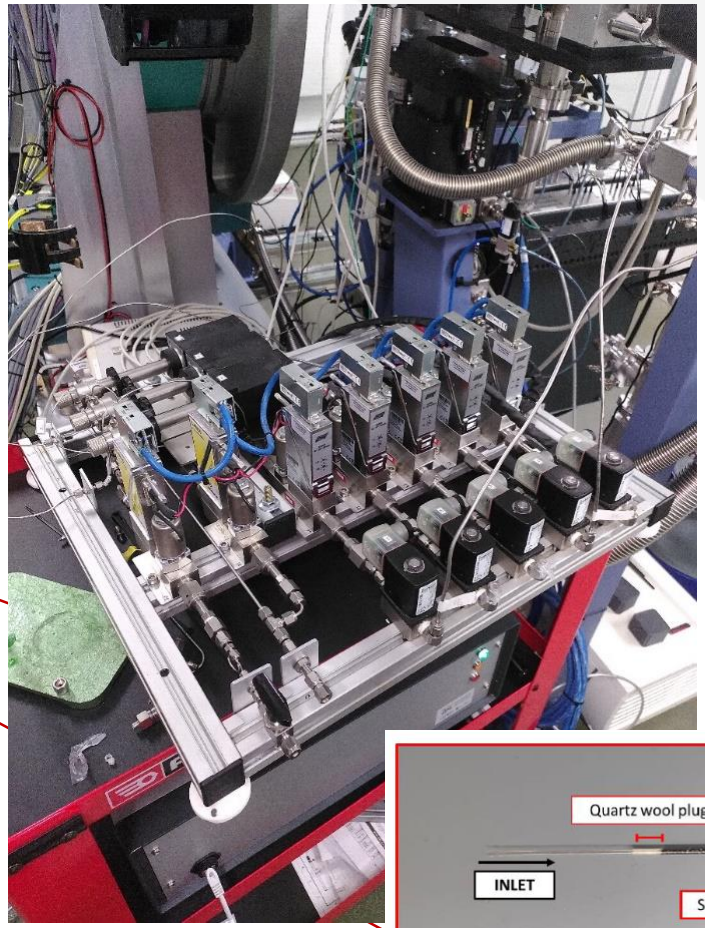
First Academic users: in situ heterogeneous catalysis

Towards the understanding of surface site requirements in metal atom trapping phenomena for the synthesis of single-atom catalysts

G. Prieto, W. Henao, E. Torregrosa, S. Gutiérrez-Tarriño, J. Hernández-Fenollosa, F. Rey, P. Oña-Burgos1 (ITQ - Valencia)

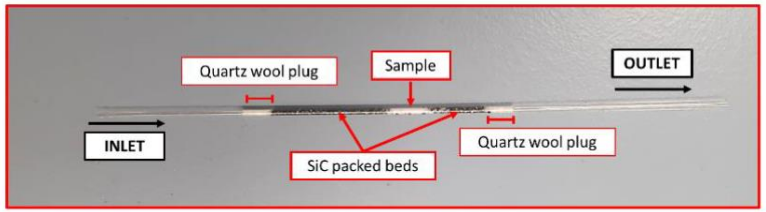




XAS in fluorescence geometry:

- Mn k edge 6539 eV
- Co K edge 7709 eV
- Ru k edge 22117 eV

<u>Spinning capillary cell</u>
Hot gun (RT-600 C)



24th – 29th of May 2022 at NOTOS

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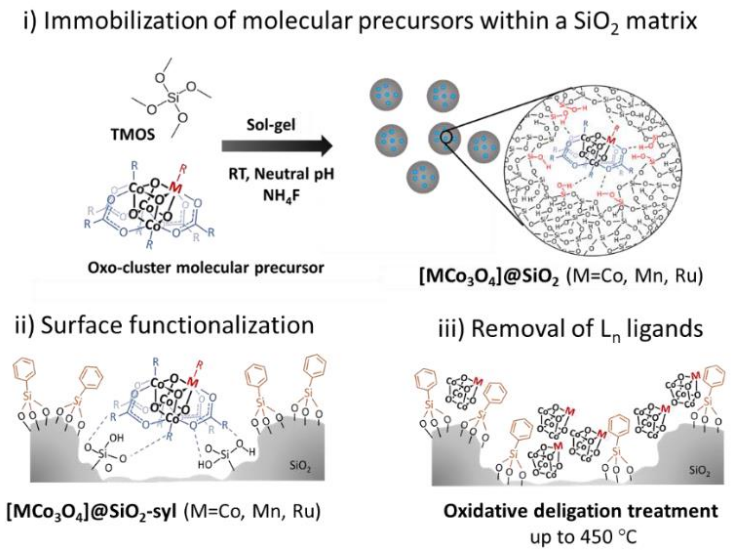
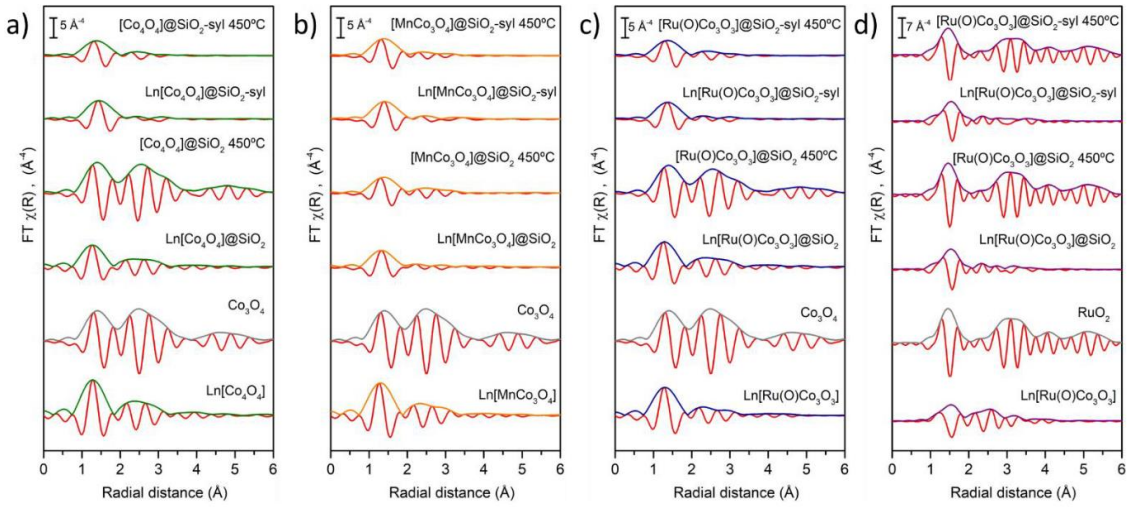
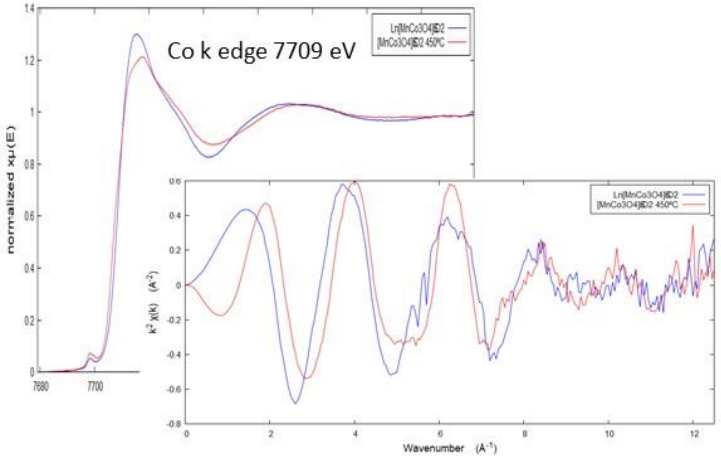
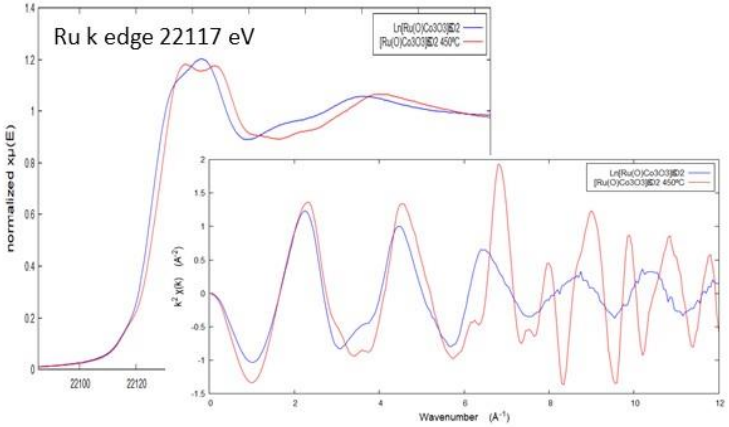
First Academic users: in situ heterogeneous catalysis

Towards the understanding of surface site requirements in metal atom trapping phenomena for the synthesis of single-atom catalysts









More details by Wilson Henao #63 AUSE 06/09/22

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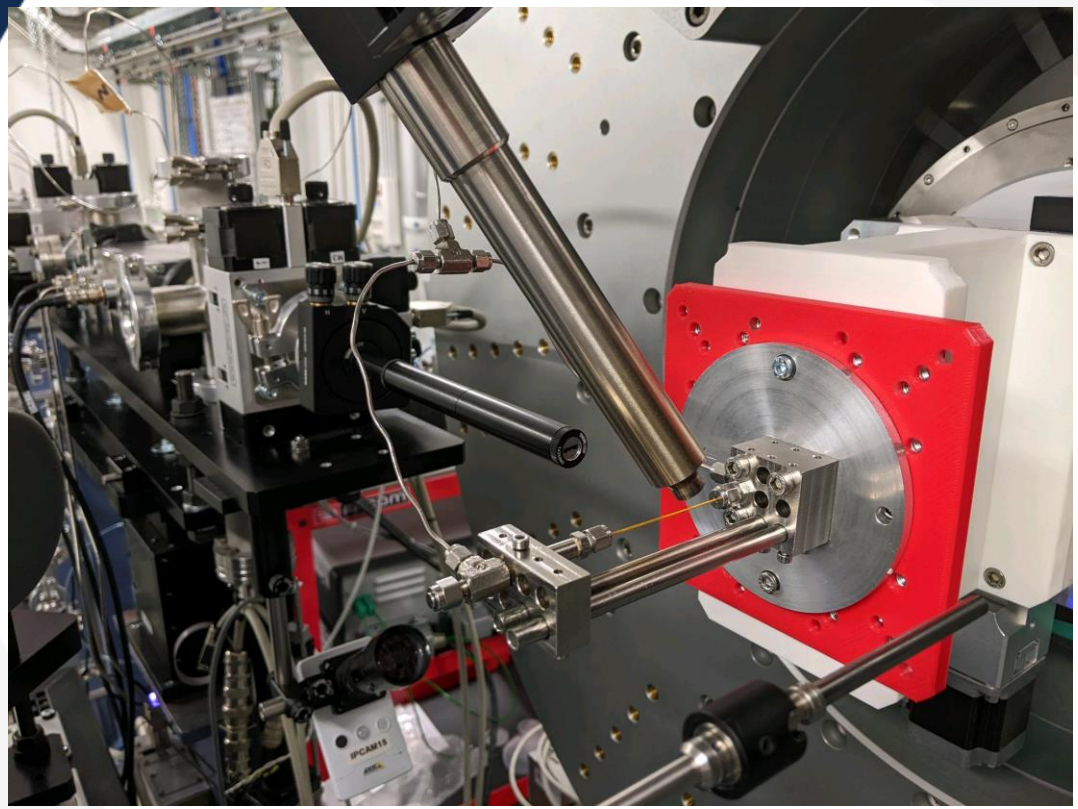
Experimental setup available at NOTOS

Capillary setup coupled with cryostream



- Capillary cell
- Oxford **cryostream** 700 series (100-350 K)
- XRD by Mythen detector

Oriol Vallcorba (Alba) – *Understanding structural flexibility in crystalline coordination compounds. Two examples of contrasting nature, a flexible Metal Organic Framework and a Porous Molecular Material*

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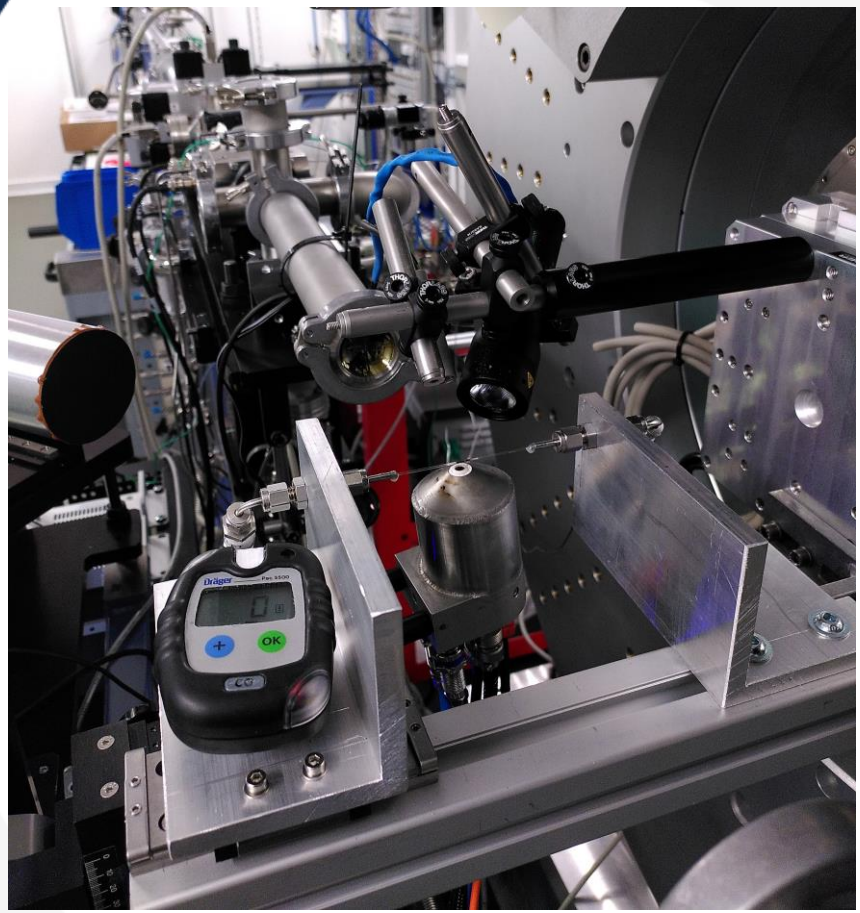
Experimental setup available at NOTOS

Capillary setup coupled with hot air blower



- Capillary cell
- **UV-vis** led
- FMB **Oxford hot air blower** (RT-900 C)
- XAS in fluorescence configuration
- XRD by Mythen detector

Ana Iglesias (CSIC - Madrid) *Investigation by XAS and XRD of Copper/Zinc catalysts for CO_2 hydrogenation*

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Experimental setup available at NOTOS

SPECAC cell for self-supported pellets

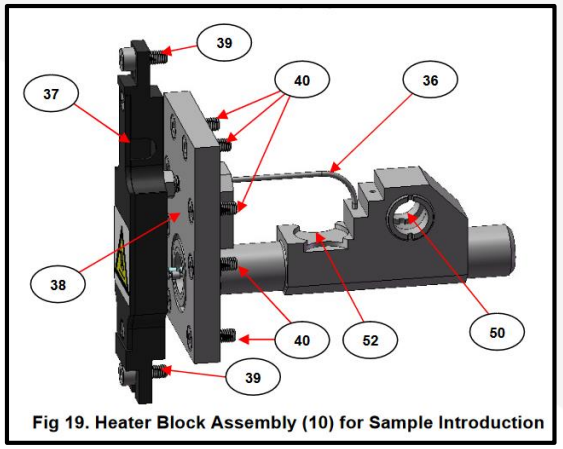

Fig 19. Heater Block Assembly (10) for Sample Introduction

- Self supported pellet
- Only XAS transmission measurement
- RT – 700 °C
- Ambient pressure

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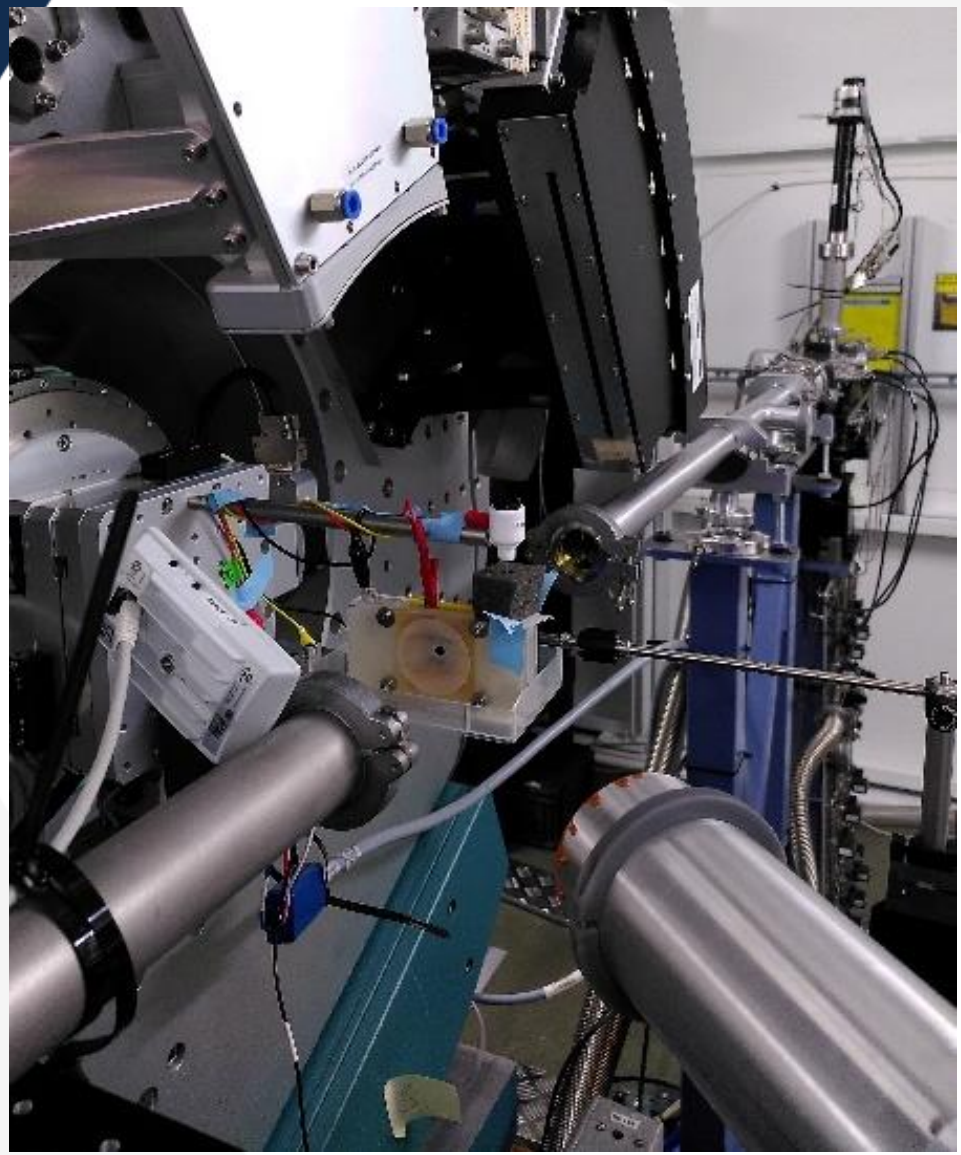
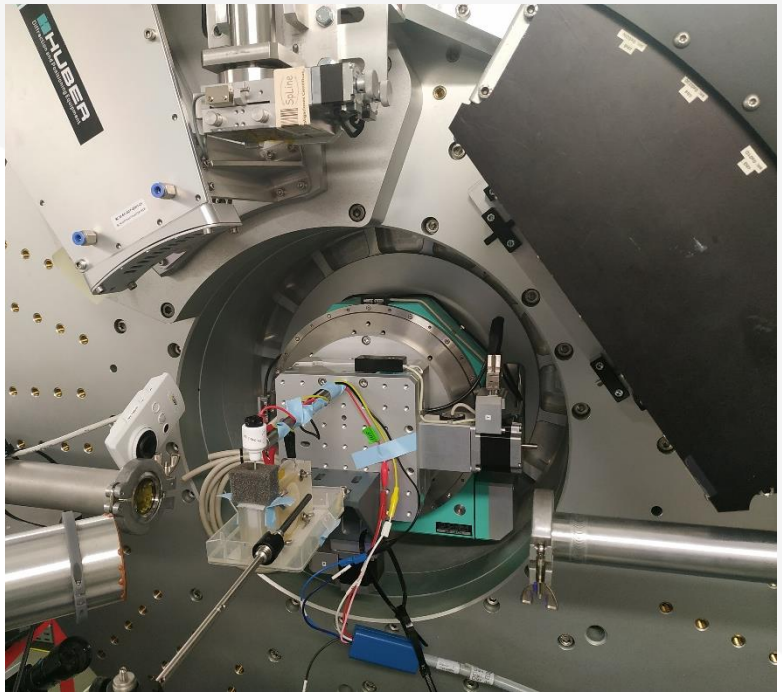
Experimental setup mounted at NOTOS

Users' setup for electrochemistry





- **Electrochemistry cell**
- XRD by Mythen
- XAS in transmission and fluorescence at Ir L_3 edge

Marianne van der Merwe ((HZB – Berlin) *Correlating potential- and pH induced transformations of highly active iridium-based electrocatalysts with catalytical stability using operando XANES and EXAFS*

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Νότος Team

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Carlos Escudero
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Beamline scientist
Hiring process on going

Jordi Prat
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Post doc
Hiring process on going

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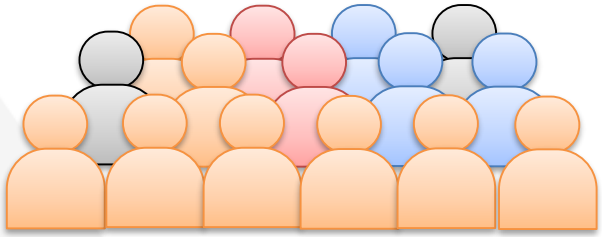
Νοτος development Team

BL Scientists	**Giovanni Agostini** **Carlos Escudero** **(Oriol Vallcorba)**
Technician	**Jordi Prat**
BL Engineer	**José Ramón Molinero**
BL electronic engineer	**Álvaro Baucells Costa**
Control System Engineer	**Roberto Homs**
Project executive	**Josep Nicolàs**

+ Project direction
+ Stakeholders
+ Admin. Support



WP BL Design	**Josep Nicolas**
WP Front-End	**Jordi Marcos**
WP BL optical components	**José Ramón García**
WP Vacuum Standard Items	**Raquel Monge**
WP Metrology station	**Dominique Heinis**
WP XAS/PD station	**Oriol Valcorba**
WP Electronics hardware	**Jose Avila**
WP Electronics infrastructure	**Alberto Ruz**
WP Systems infrastructure	**Toni Pérez**
WP Controls	**Roberto Homs**
WP EPS and PSS	**Nil Serra**
WP Rad. Shielding	**Núria Martí**
WP Services and control hutch	**Núria Martí**
WP CSN license	**Maria Jose Garcia Fuste**
WP Transport and moving	**Antoni Camps**
WP Tunnel coord.	**Montse Pont**
Workshop	**Jose Ferrer**

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Gases @ NOTOS

Nr	Gas[1]	Max Flow (ml/min)	P range (bar)[2]
1	**Hydrogen**	50	Patm - 20
2	**CO**	50	Patm - 20
3	**Methane**, ethane, propane	50	Patm - 20[3]
4	**Ethylene**, propylene	50	Patm - 9[4]
5	**He**	200	Patm - 20
6	**Ar**	10	Patm - 20
7	**CO_2**, N_2	50	Patm - 20
8	**Oxygen 20%**	50	Patm - 20
9[5]	NO ≤ 2%; NO_2 (+21% O_2) ≤ 0.5%; N_2O ≤ 36%	50	Patm - 5
10[5]	NH_3 ≤ 25%	50	Patm - 5

1: For not dedicated gas lines, gas for calibration is indicated in **bold** letters. All dilutions are done in He.

2: No pressure control from Patm to 4bar. In a second stage of the gas system design it is planned to implement a back-pressure controler up to 80 bar.

3: Up to 6 bar for propane

4: Up to 8 bar for propylene

5: Gas for calibration: He

More information document session of the website:
https://www.albasynchrotron.es/en/beamlines/bl16-notos

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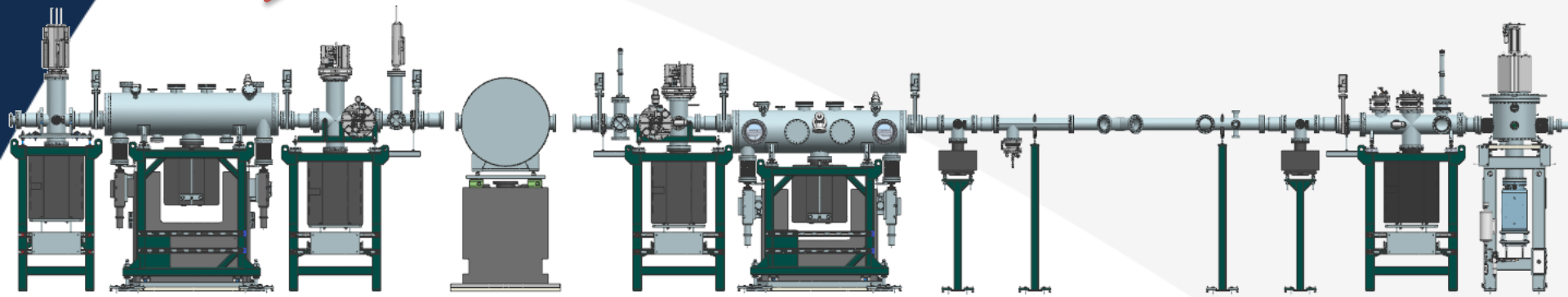
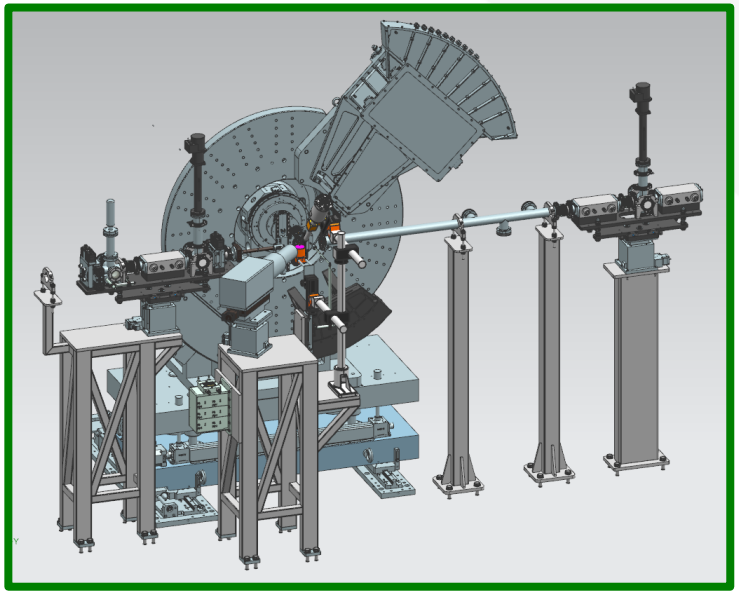
NOTOS schedule

Beam Direction



- **FE** commissioning with X-ray: 9th of September 2020
- First **X-ray** in **OH**: 12th of February 2021
- **X-ray** at **sample** position in PD&XAS station: 12th of July 2021
- **BL rea**dy for X-ray (**with M2**): 4th of March 2022
- X-ray commissioning **SDD**: 10-14th May 2022
- **First Expert Users**: **26th of April 2022**
- **First Academic User**: **24th of May 2022**
- **Multipurpose** endstation: Autumn 2022
- System for **reactive gas** available: Autumn 2022



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