In Situ **C**orrelative Facility

for **A**dvanced **E**nergy **M**aterials

Catalan branch of the Advanced Materials Programme of the Complementary R&D&I Plan

L. Aballe

Finançat per:
Unió Europea Fons Europeu Next Generation
Gobierno de España
Plan de Recuperación, Transformación y Resiliencia
Next Generation Catalunya
Generalitat de Catalunya Departament de Recerca i Universitats

In Situ **C**orrelative Facility for **A**dvanced **E**nergy **M**aterials

In-CAEM will develop a **singular infrastructure** for research in **advanced energy materials** in order to address the scientific challenges of the European Green Deal:

Multi-modal
Multi-lengthscale
Correlative

in situ/operando experiments, combining

- **(Scanning) transmission electron microscopy**
- **Scanning probe microscopies**
- **Synchrotron X-rays** (spectroscopy, diffraction,…)
- **Advanced data analytics** (HPC, deep learning, AI,…)

Open to all the scientific community.

Mix & Match techniques and methodologies to tackle complex problems

Single entry point: ALBA User office

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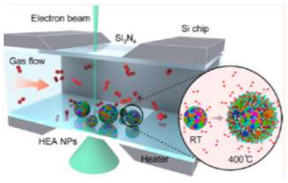
WP1: (Scanning) Transmission Electron Microscope

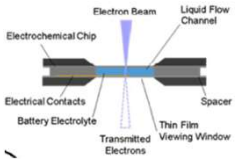
J. Arbiol (ICN2), L. Aballe (ALBA), B. Ballesteros (ICN2), J. Oró (ICMAB)

149 person*month (30% staff)

Aberration corrected (S)TEM	30-300 keV, 4D STEM, EDX, EELS, tomography, ptychography, …
Catalysis sample holder	Controlled gas mixing and flow, HV-2 bar, RGA product analisys, controlled T ramps
Electrochemistry sample holder	Liquid Flow, In situ electrodes, Potentionstat
Variable temperature sample holder	Liquid nitrogen cooling, heating, biasing

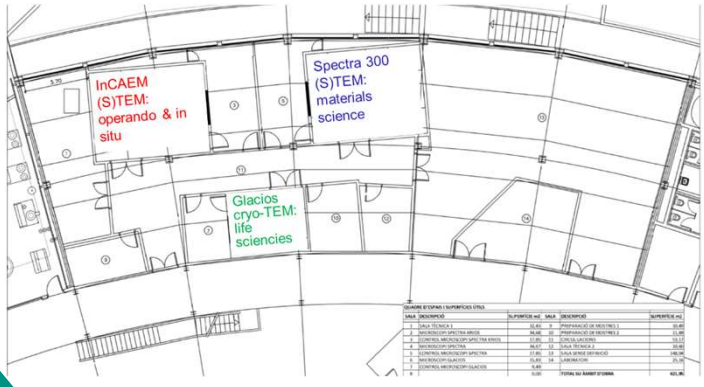


Song et al, ACS Nano (2020)



Singh et al, ACS App. Ener. Mat. (2020)

JEMCA: Joint Electron Microscopy Center at ALBA



Pablo Guerra (IBMB) -cryo TEM 

Jordi Arbiol (ICN2) 

Belén Ballesteros (ICN2) 

Bernat Mundet (ICN2) – METCAM operator 

Kapil Gupta (ICN2) – METCAM operator 

Alba Garzón-Manjón (ICN2) – in situ STEM 

Sara Martí (ICN2) – ReMADE@ARI 

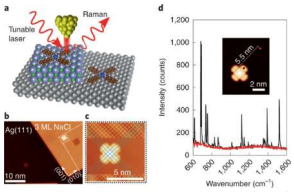
Núria Bagués (ALBA) – in situ STEM 

WP2: Scanning Probe Microscopes

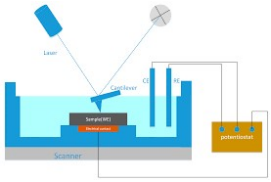
A. Mugarza (ICN2), C. Ocal (ICMAB), E. Barrena (ICMAB)

191 person*month (19% staff)

UHV Photon SPM	STM/nc-AFM with integrated preparation chamber for in-situ sample preparation, operating in UHV and cryogenic temperatures, with optical input/output access for photon spectroscopies (TERS, TEPL, STML, Tunneling photocurrent…)
Environmental Photon SPM	AFM with capability for optical spectroscopies (TERS, TEPL, co-localized Raman…), operated in controlled gas and liquid environments
UHV SPM (existing)	STM/nc-AFM system operating in UHV and 300K, with integrated preparation chamber for in-situ sample preparation.
Multipurpose SPM	Multipurpose and multimodal AFM (KPFM, EFM, CFM…), operated in controlled humidity, gas and liquid environments (sealed or perfusion)

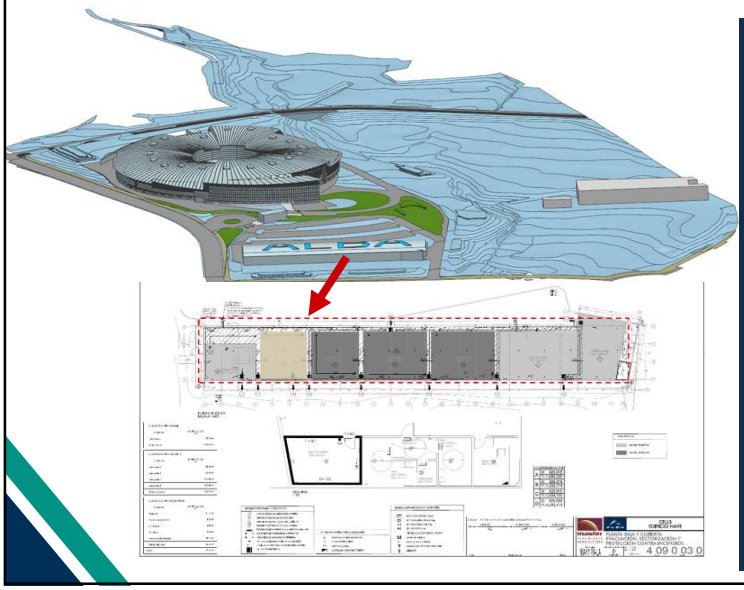


Jaculbia et al, Nat. Nano. 15 (2020)



Chen et al, Materials 13 (2020)

Scanning Probe Microscopy platform at ALBA



Carmen Ocal (ICMAB)

Esther Barrena (ICMAB)

Alba Cazorla (ICMAB) - AFM operator

Rogger Palacios (ICMAB)
- STM/AFM postdoc

Daniel Martín (ICMAB) - AFM scientist

Marc G. Cuxart (ICN2)
- UHV Photon SPM scientist

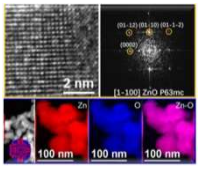
Aitor Mugarza (ICN2)

Emigdio Chavez (ICN2) – STM/TERS scientist

WP3: BL adaptations for correlative experiments

L. Aballe (ALBA), J. Nicolás (ALBA), C. Frontera (ICMAB)

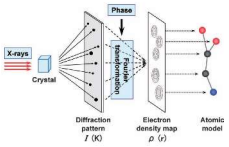
134 person*month (47% staff)



Multimodal nanocaracterization

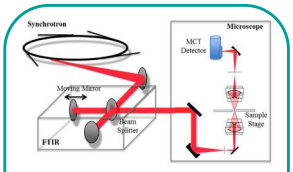
(S)TEM

X-ray diffraction
NOTOS/MSPD



Atomic structure crystalline materials

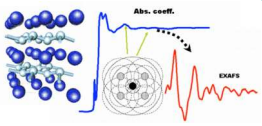
- Portable gas & liquid micro-reactors
- Adapter stages in beamlines
- Microfocusing



Vibrational modes, phonons. Organic and light molecules.

IR microscopy
MIRAS

X-ray absorption spectroscopy
NOTOS

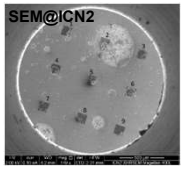


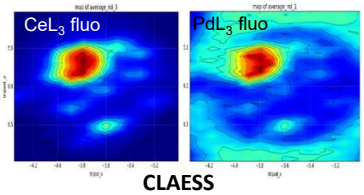
Composition, valence, coordination.
Any aggregation state.

Feasibility tests

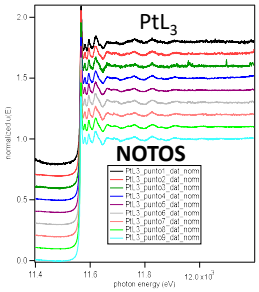
6 beamlines, 4 institutions (ALBA, ICN2, UPC, ICMAB)

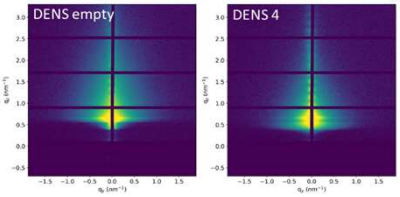
Catalysis sample: Pt/Pd NPs on CeO_2 NPs matrix (N. Divins, UPC)





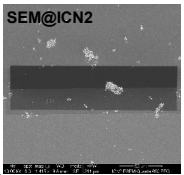
CLAESS

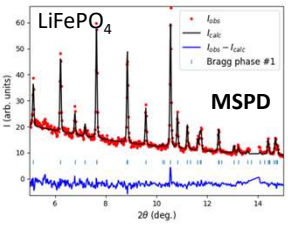


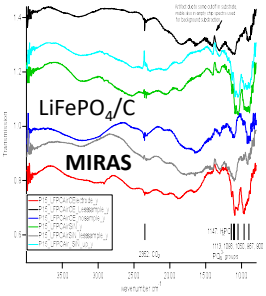


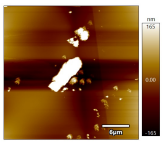
NCD-SWEET

Electrochemistry sample: $LiFePO_4$ (R. Palacin, ICMAB)

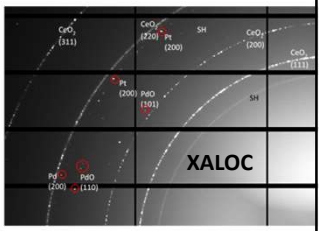




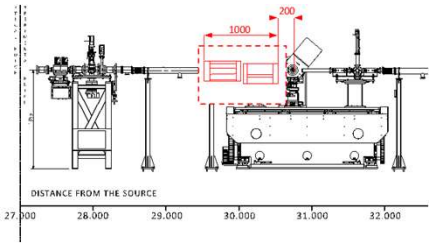


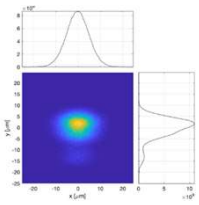


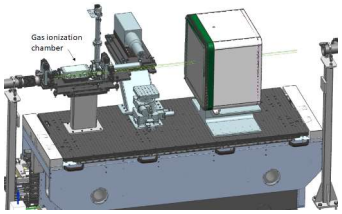
AFM@ICMAB

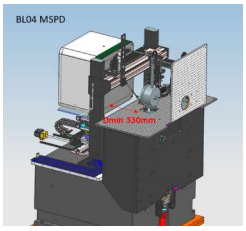


WP3: BL adaptations for correlative experiments









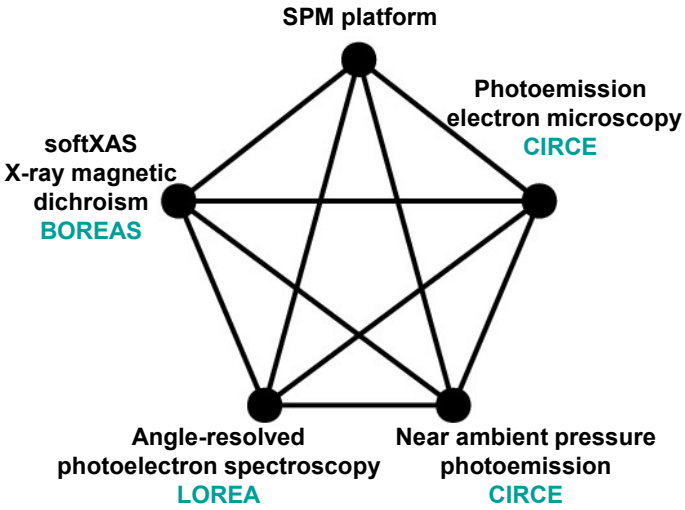
- µNOTOS
- XRD with PILATUS detector @ NOTOS & MSPD

Interfaces X-rays/EM group leader:
Sandra Ruiz-Gómez (ALBA, 2024)

Mechanical & micro-reactors engineer :
Antoni Garcia de Herreros (ALBA)

2x Micro-reactors research technicians:
Soon open!

WP3: BL adaptations for correlative experiments



X-ray (dichroic) absorption spectroscopy
BOREAS

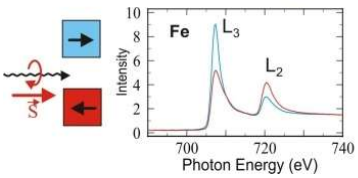
Element-resolved magnetization

Spin/orbit contributions

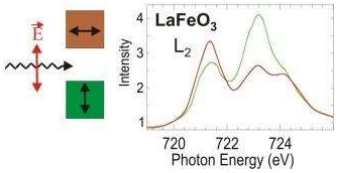
Variable fields and temperatures

Sub-ML surface sensitivity

Circular Dichroism - Ferromagnets



Linear Dichroism - Antiferromagnets



J. Stohr, NEXAFS Spectroscopy

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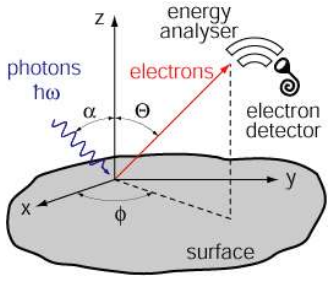
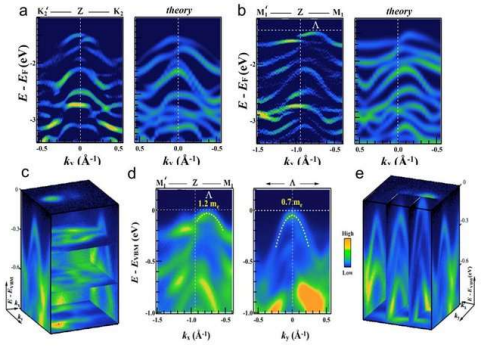
Angle-resolved photoelectron spectroscopy
LOREA BL

(Spin-resolved) band structure of solid materials E(k)



Photoelectrons conserve information about the energy and wavevector they had in the sample.

Photoemission intensity vs energy and angle



$ReSe_2$. Hart, et al., Sci. Rep. 7 (2017)

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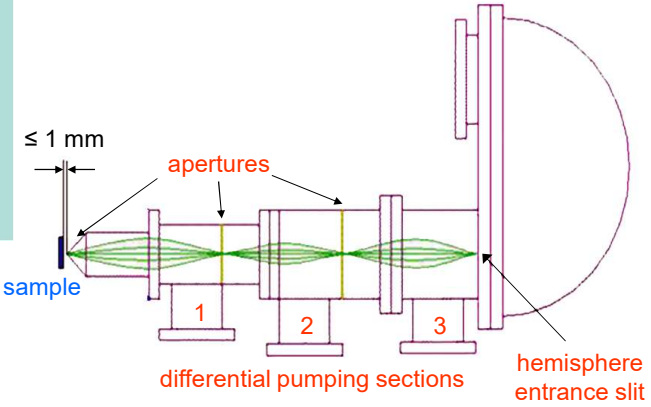
Near ambient pressure photoemission CIRCE

Core level photoemission in mbar pressures (& gas pase!)

Depth profiles changing photon energy

Sub-ML surface sensitivity



Salmeron (ALS) & Schlögl (FHI)

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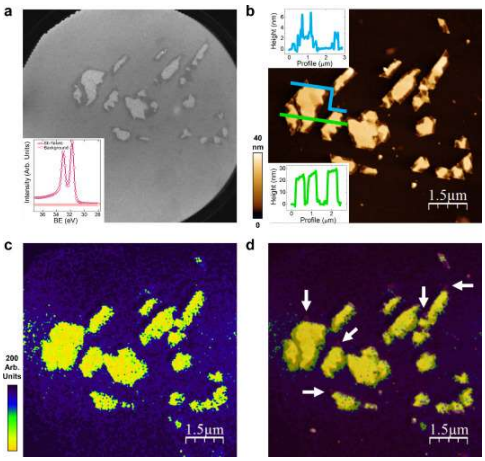
X-ray photoemission electron microscopy CIRCE

Chemical, electronic, and/or magnetic contrast

Surfaces, thin films & nanostructures

Sub-ML surface sensitivity

Multi-technique instrument (uLEED, LEEM,…)



Ares, et al., App. Mat. Today 24 (2021) 101132
Few-layer antimonene electrical properties..

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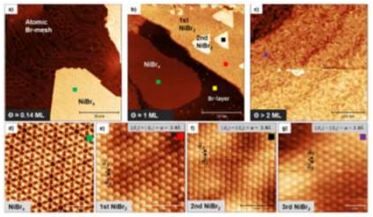
Multi-modal analysis 2D materials

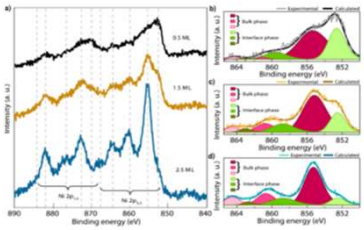
Noncollinear Magnetic Order in Two-Dimensional $NiBr_2$ Films Grown on Au(111)

Djuro Bikaljević,* Carmen González-Orellana, Marina Peña-Díaz, Dominik Steiner, Jan Dreiser, Pierluigi Gargiani, Michael Foerster, Miguel Ángel Niño, Lucía Aballe, Sandra Ruiz-Gomez, Niklas Friedrich, Jeremy Hieulle, Li Jingcheng, Maxim Ilyn,* Celia Rogero, and José Ignacio Pascual*

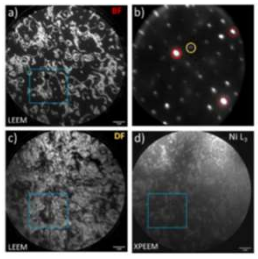
Cite This: *ACS Nano* 2021, 15, 14985−14995 Read Online



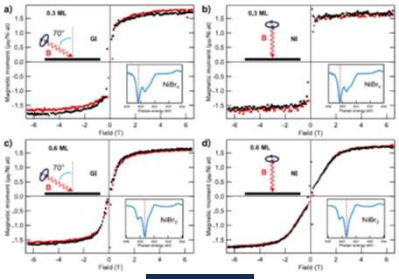
STM



XPS

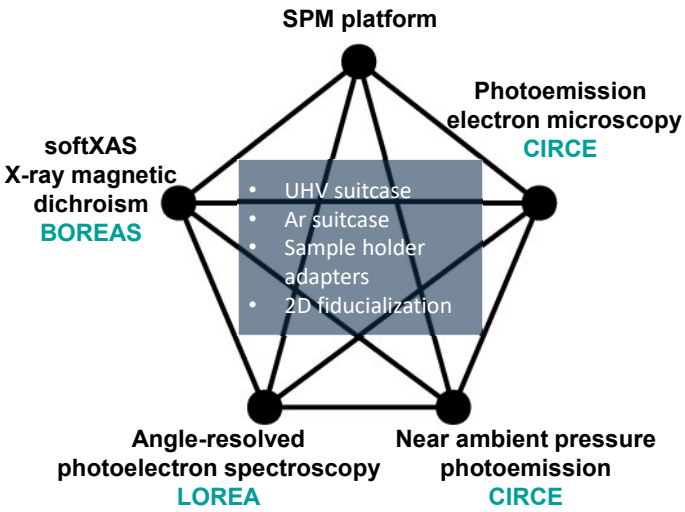


LEEM/LEED/XPEEM



XMCD

WP3: BL adaptations for correlative experiments



WP4: Infrastructure & methods for data analysis

G. Merino (PIC), P. Ordejón (ICN2), A. García (ICMAB), M. Eriksen (PIC), F. Torradeflot (PIC), V. Acín (PIC)

81 person*month (40% staff)

WP5: Infrastructure & methods for in situ data analysis

S. Vicente (ALBA), A. García (ICMAB), J. Otón (ALBA), N. Soler (ALBA), G. Rosas (ALBA)

132 person*month (18% staff)

Development of methodologies:

- Automatized workflows for analysis of large datasets
- High throughput, combinatorial studies
- Multimodal analysis
- Multi-lengthscale analysis in 2D and 3D
- Machine learning supported pipelines
- Simulations

Multimodal methodology :
Oleg Usoltsev (ALBA)

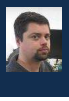
Industrial researcher – alloys:
Leila Panahi (ALBA)

Multilengthscale methodology: ongoing

Integration engineer:
Andrés Fraile (ALBA)

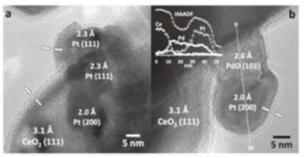
Scientific data engineer:
Przemyslaw Karczmarczyk (ALBA)

AI Methodology scientist:
Martin Eriksen (PIC-IFAE)

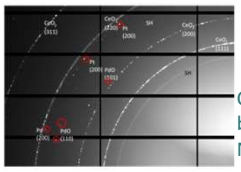


Correlative workflows

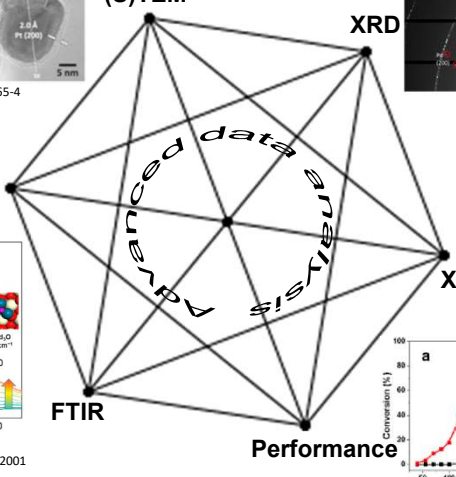
Are these the active NPs in reaction conditions?



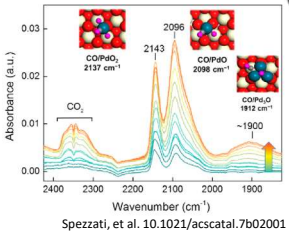
Divins, et al. 10.1038/s41467-022-32765-4



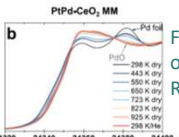
Can we follow the behavior of individual NPs/microcrystals?



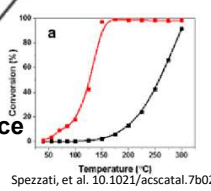
Reaction intermediates? Active sites?



Spezzati, et al. 10.1021/acscatal.7b02001



Fraction and nature of amorphous phases? Role of substrate?



Spezzati, et al. 10.1021/acscatal.7b02001

WP6: Infrastructure

J. Casas (ALBA), C . Orozco (ALBA), N. Martí (ALBA)
45 person*month (100% staff)

- STEM lab conditioning
- New STEM sample preparation space
- SPM platform lab building

WP7: Outreach

A.B. Martínez (ALBA), A. May (ICMAB), A. Argemí (ICN2), S. Grinschpun (IFAE)
8 person*month (100% staff)

- Ciencia Viva @ CCCB (together with other PPCC projects where Catalunya participates)

InCAEM Talks

Date	Time	Speaker	Talk
01/03/2023	12:00	Alba Garzón Manjón	In-situ heating (scanning) transmission electron microscopy for exploring the thermal stability of a nanoscale complex solid solution thin film
14/04/2023	09:00-16:00	InCAEM Workshop	Planes Complementarios on Advanced Materials in Catalonia
03/05/2023	12:00	Juan Jesús Velasco Vélez	Correlative in situ scanning electron microscopy / X-ray spectroscopy characterization of electrochemical interfaces
07/06/2023	12:00	Daniel Martin Jiménez	Atomic Force Microscopy: From Liquid to Ultra-High Vacuum Environments
14/07/2023	12:00	Ningyan Cheng	In-situ Transmission Electron Microscopy Study of Functional Nanomaterials
06/09/2023	12:00	François Fauth	Synchrotron powder diffraction: from the basic theory to in Situ experiments on operando systems
04/10/2023	12:00	Martin Eriksen	AI-Driven Material Science: InCAEM and the Port d'Informació Científica (PIC) Data Center

WP8: Coordination, exploitation model

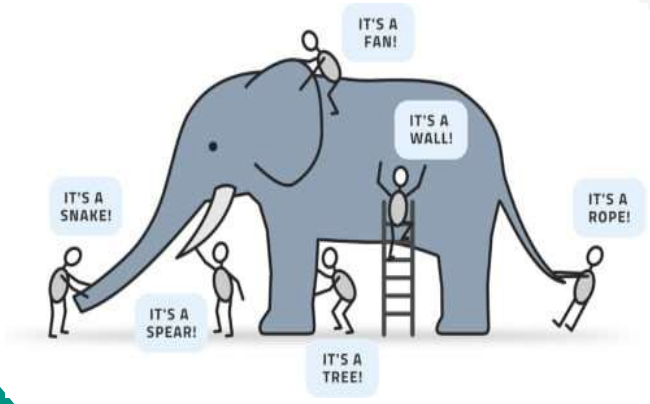
C. Biscari (ALBA), J. Arbiol (ICN2), M. Pont (ALBA)
13 person*month (100% staff)

New Access modes:

- Multimodal
- Resident users?

Long term sustainability

Thank you!



https://www.cells.es/en/instrumentation/incaem

laballe@cells.es

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