



## STORAGE RESEARCH INFRASTRUCTURE ECO-SYSTEM

### RI Information sheet 2022

#### CIC energigUNE, Solid state NMR platform (EES-TES)

Solid-state NMR can provide very accurate information about the structure and the dynamics of molecules and ions in electrochemical materials. This is crucial for the understanding and the development of energy storage devices. Solid-state NMR is a sensitive technique to short-range order that surpasses traditional techniques of materials characterization as X-ray diffraction or electron microscopy in the study of amorphous materials and complements these techniques in the study of crystalline materials. Furthermore, solid state NMR can provide dynamic information with atomic specificity in highly disordered and even fully amorphous materials.

The technologies of the solid-state NMR platform at the CIC energigUNE are specially designed for the characterization of the materials used for energy storage including in-situ measurements during device operation and variable temperature studies (-120 C degrees to +400 C degrees).

The solid-state NMR platform at CIC energigUNE was recognized in 2018 by Bruker-Spain as the reference center in solid-state NMR in the Spain.

The main area of specialization of the platform is focused on materials used in energy storage devices including paramagnetic cathode materials, metallic and carbon-based anodes and solid electrolytes (polymers, ceramics and composites). The analytical services include the analysis of pristine materials, additives and formulations. These analyses are also performed in-situ to understand the phase transitions and dynamic processes occurring during battery operando and the post-mortem studies of batteries and supercarps.



Additionally, our platform is strongly involved in the characterization of thermal energy storage materials. In addition to energy storage materials, our capabilities have been used in the characterization of polymer materials and biological systems. The knowledge obtained by the solid-state NMR characterizations are crucial for the rational designed of improved materials and therefore key in high-quality research.

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<b>Project Acronym</b>	StoRIES
<b>Call</b>	H2020-LC-GD-2020
<b>Grant Agreement No.</b>	101036910
<b>Project Start Date</b>	01-11-2021
<b>Project End Date</b>	31-10-2025
<b>Duration</b>	48 months



## 1. Photo



## 2. Geographical coordinates (°, ′, ... N/S, E/W)

42.911162, -2.668712

## 3. Description of the research infrastructure for the webpage

The solid-state NMR platform at CIC energiGUNE is composed by state-of-the-art equipment specially designed for the analysis of energy storage materials including batteries, supercaps and thermal energy store materials also by ex situ and in situ methods. We have a long record of publications, industrial projects and international collaborations in these fields. In particular, our platform is composed by:

### 1. WB BRUKER AVANCE III 500 MHZ

The Avance III 500 MHz was installed at CIC in April 2012. It is a wide bore magnet especially suited for non magnetic materials where high resolution is desired.

WB This high field spectrometer is designed for the characterization of solid materials for electrochemical energy storage applications. Including anode, cathodes, ceramic and polymer-based solid electrolytes and supercaps. These materials can be studied ex-situ and in-situ using a custom-made probehead that is not accessible in common solid-state NMR laboratories. Our areas of expertise include the structural characterization of battery materials, the understanding of the degradation mechanisms and post-mortem analysis of batteries and supercaps, the understanding of the key parameters involved in the observed electrochemical performance and the kinetic processes of Li<sup>+</sup> and Na<sup>+</sup> diffusion.

The materials investigated include ceramics, polymers, metals and alloys, porous materials, biologic materials and composites. All these areas are open to external users. The in-situ system is the phase of testing and implementation and is expected to be available to external users by March 2021.

In the area of thermal energy storage, our platform is equipped with variable temperature probes that allow the in-situ characterization of phase transitions in organic and inorganic materials up to 200C.

#### Available Probes:

- Double resonance  $1\text{H}/19\text{F-X}$  DVT CPMAS 1.3 mm probe that can reach ultra-fast spinning speeds of up to 67 kHz
- Triple resonance  $1\text{H-X-Y}$ , DVT CPMAS 2.5 mm probe that can spin up to 35 kHz
- Static wideline H-X probe for variable temperature studies
- Variable temperature measurements between 300°C and -100°C

## 2. WB BRUKER AVANCE III 200 MHZ

The Avance III 200 MHz was installed at CIC in March 2012. It is a wide bore, low magnetic field magnet dedicated to the study of paramagnetic materials which are a frequent component in battery and supercapacitor electrodes.

This low field solid state NMR spectrometer is specially designed for the characterization of paramagnetic materials that are used as cathode electrodes. This magnet combines a rather low magnetic field with a very fast MAS probe of 1.3 mm rotors allowing Magic Angle Spinning (MAS) frequencies of up to 67 kHz. This setup is rather unique and is essential for the accurate characterization of cathode materials. We have a very strong background of high impact publications in this field and we are among the few solid-state NMR platforms that are able to perform such measurements worldwide.

#### Available Probes:

- Double resonance  $1\text{H}/19\text{F-X}$  DVT CPMAS 1.3 mm probe that can reach ultra-fast spinning speeds of up to 67kHz.
- Double resonance  $1\text{H-X}$  DVT CPMAS 4 mm probe, that can reach temperatures up to 400°C and spinning speeds up to 18 kHz.

- Variable temperature measurements between 400°C and -100°C.

Our solid-state NMR platform was officially recognised in 2018 by Bruker-Spain as the reference centre for solid state NMR in Spain. Our platform combines state of the art hardware and software capabilities with a highly qualified human resources for the experimental design and deep analysis of the solid-state NMR data.

#### 4. Availability of the research infrastructure

(Please indicate time periods in which infrastructure will not be available for StoRIES in the next 2 years – if already known)

To be checked with CIC energiGUNE's contacts.

#### 5. Special considerations (confidentiality / NDA agreements, insurance requirement, special training, HSE training)

##### **Modality of access under this proposal:**

All our solid-state NMR capabilities are open to external users. The access to the platform can be provided through the incorporation of trained users for direct use of the equipment prior approval of the platform manager (as users should demonstrate their expertise in the field before directly using this advanced equipment). Alternatively, users can send their samples to be analysed and characterized by our trained personnel.

For users interested in the direct use of the spectrometers, an NMR training will be offered in our platform. Once the users get the approval of the platform manager, they will be allowed to access the installations under the supervision and support of the platform manager. In any case, the nature of the materials investigated and the experiments planned should be communicated to, and approved by the platform manager in order to exclude any potential hardware or health risks.

##### **Confidentiality / NDA agreements, insurance requirement:**

As contemplated inside the general rules and agreements of StoRIES.

The users -should need it and required from their side, will contact CIC energiGUNE to arrange NDA prior to the organization of the direct use or service.

### Review procedure under this proposal:

According to the rules of STORIES, users will be selected through the Selection Committee, which will evaluate the proposals coming from the Academia, RTOs and Industry.

6. Energy storage technology that can be analysed/studied by using the research infrastructure

- Electrochemical
- Chemical
- Thermal
- Mechanical
- Superconducting Magnetic
- Cross-cutting  (Specifically:...)

7. Key words for the webpage

Solid state NMR, Paramagnetic solid-state NMR, *in situ* NMR, material characterization.

8. TRL level (if applicable):

- 1-3
- 4-6
- Above