

## MAGNETIC RESONANCE FOR BATTERIES RESEARCH AND MANUFACTURING

# Battery research probes for Li-ion technologies (and beyond)

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*Innovation with Integrity*

### Introduction

At the core of electrification strategies, rechargeable batteries are key to enabling the successful global transition to renewable energy sources, helping to balance grids and reduce loss. To address the fast-growing demand for ever more advanced products to meet ambitious global net zero emission targets, battery manufacturers and researchers are ramping up R&D activities and production capacity. In essence, they need to adopt technologies that will enable them to deliver innovative, economical and high-quality solutions with high performance on limited environmental footprints and short lead times. One solution that will help to realize these goals is in situ solid-state nuclear magnetic resonance (NMR) spectroscopy. By harnessing the power of state-of-the-art probes and consoles, subject matter experts can benefit from optimized R&D as well as rapid and accurate quality inspection activities.

Batteries are ubiquitous technologies, as they support multiple, diverse applications. The highest demand with double digit annual growth rates is projected for the Battery Electric Vehicle (BEV) market whilst the second biggest market sector for electro-chemical energy storage will be for Stationary Storage with grid buffering and off-grid applications. As batteries offer a key solution for energy storage, they are considered an enabling technology for the effective transition towards more sustainable, renewable energy sources, able to help maximize the use of energy that can be generated from cyclic sources such as solar and wind.

Large scale industrial battery production is requesting electro-chemically active material at high amounts and low cost, with safe supply chains, and of course, an electro-chemical performance supporting top products. Safety, service life, power density and recharge efficiency of batteries are all defined by the physical and chemical properties of their components, namely their cathodes, anodes, separators and electrolytes. Therefore, R&D investigations and quality inspections are key. In effect, understanding electrochemically active materials and their interaction is a mainstay in academic and translational research, industrial R&D, quality assurance/quality control (QA/QC) as well as product innovation, e.g. new electrode and electrolyte materials.

More precisely, these analyses are fundamental to developing a better understanding of what attributes drive quality and performance, determining how these can be designed into products through suitable processing conditions as well as advancing the capabilities of next-generation battery systems. Moreover, detailed characterizations can help improve manufacturing efficiency, reducing cost, waste, rework, energy and raw material use.

Solid-state NMR is a powerful and versatile tool to gain insight into the structural changes of battery materials and to explore and research new materials. While most of these investigations are performed ex situ a number of key questions to new battery applications arise from working conditions; i.e. when a battery is charge/discharged. To effectively measure these processes and critical quality attributes, analytical solutions for the characterization of batteries should be able to operate in situ. NMR spectroscopy offers an ideal analytical method to address the need for accurate, responsive, in situ detection in the battery sector.

### The capabilities of NMR spectroscopy

This analytical method, applied as a solid-state technique for battery analysis, can provide detailed qualitative and quantitative molecular-level insights while being non-destructive and non-invasive. In effect, it supports the detection of amorphous and crystalline species/products within the entirety of all battery components, as long as the chemical species to investigate are containing NMR active nuclei, e.g.  $^7\text{Li}$ ,  $^{23}\text{Na}$ ,  $^{13}\text{C}$ ,  $^{31}\text{P}$ ,  $^1\text{H}$ ,  $^{19}\text{F}$ , and many more.

As a result, in situ NMR spectroscopy can provide a thorough description of the chemical and electrochemical processes taking place and resulting reaction products in real time. In particular, it is possible to extrapolate information on stable and transient chemical species being produced within the materials and interfaces of battery cells, their concentration and mobility as well as reaction rates.

Current in situ NMR analyses focus on gaining detailed, accurate insights into the structure and dynamics of Li-ion battery materials as well as potential Na-ion alternatives and beyond Li-ion battery technologies, e.g. potassium and magnesium batteries.

### State-of-the-art NMR hardware for battery research

Bruker, the market leader in magnetic resonance spectroscopy instruments, offers a comprehensive product range to support advanced, value-adding in situ solid-state NMR equipment for battery and energy storage research including static in situ NMR probes, tools, and accessories.

By leveraging an analytical setup that combines the cutting-edge RF generation console AVANCE NEO, together with technologies from Bruker's partner ePROBE, subject matter experts can gather meaningful insights. The portfolio of compatible products includes probes with specialized in situ plastic cell capsules that support different configurations, i.e. single resonance (X) or double resonance (H-X or F-X) probes with automatic calibration and setup features. Each probe is equipped with up to three highly-shielded current collector ports to allow the charging and discharging of electrochemical cells while performing the NMR experiment. Furthermore, the probe is extendable with integrated flow channels for gases or liquids to effectively conduct investigations into redox-flow and metal-air batteries or similar systems.



Figure 1 front of the probe

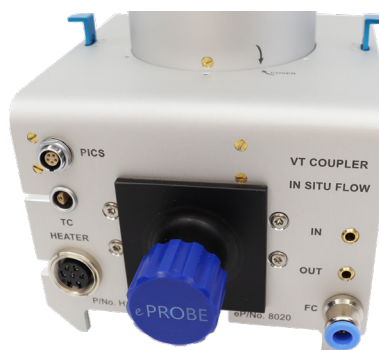


Figure 2 back of the probe

In addition to these hardware components, users can benefit from additional bespoke accessories for battery investigations, such as cells, assembly tools and the eCAT software for NMR spectroscopy data processing and visualization. All these elements can help gather key, easily interpretable quantitative and qualitative results to generate holistic, immediate product and process understanding when it comes to existing and innovative battery technologies.

## Conclusions

Rechargeable batteries and energy storage technologies are experiencing a surge in demand and increasing popularity as they offer a sustainable and efficient solution for powering a multitude of devices and storing energy from renewable sources. These solutions are therefore vital to creating a greener and more resilient future.

While current batteries already offer key capabilities, as their role as enabling technologies for the energy transition continues to grow, their performance, safety, efficiency, capacity and service life need to be improved. Even more, to meet market needs, manufacturers need to optimize their production processes, improving throughput, speed, efficiency and resource utilization. In-depth investigations are fundamental to addressing these challenges and techniques such as solid-state in situ NMR spectroscopy offer a reliable, comprehensive analytical tool.

By applying our complete, high-quality, customizable solutions, such as the combined Bruker and ePROBE offering, subject matter experts can benefit from reliable NMR setups that have been specifically developed to meet the needs of battery research within academia and industry.

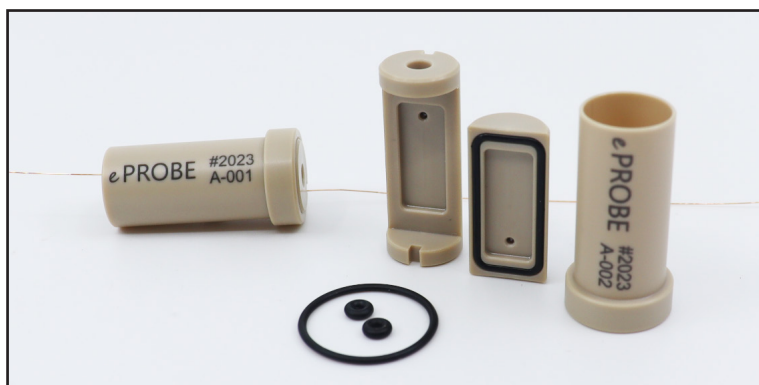


Figure 4 in situ NMR accessories (a cell)

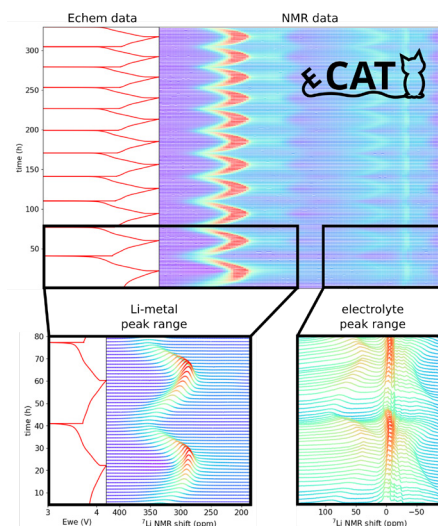


Figure 3 eCAT data

## References:

1. Kitada, K., Pecher, O., Magusin, P. C., Groh, M. F., Weatherup, R. S., & Grey, C. P. (2019). Unraveling the reaction mechanisms of SiO anodes for Li-ion batteries by combining in situ  $^7\text{Li}$  and ex situ  $^7\text{Li}/^{29}\text{Si}$  solid-state NMR spectroscopy. *Journal of the American Chemical Society*, 141(17), 7014-7027.
2. Pecher, O., Carretero-González, J., Griffith, K. J., & Grey, C. P. (2017). Materials' methods: NMR in battery research. *Chemistry of Materials*, 29(1), 213-242.
3. Pecher, O., Bayley, P. M., Liu, H., Liu, Z., Trease, N. M., & Grey, C. P. (2016). Automatic Tuning Matching Cyclor (ATMC) in situ NMR spectroscopy as a novel approach for real-time investigations of Li-and Na-ion batteries. *Journal of Magnetic Resonance*, 265, 200-209.



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