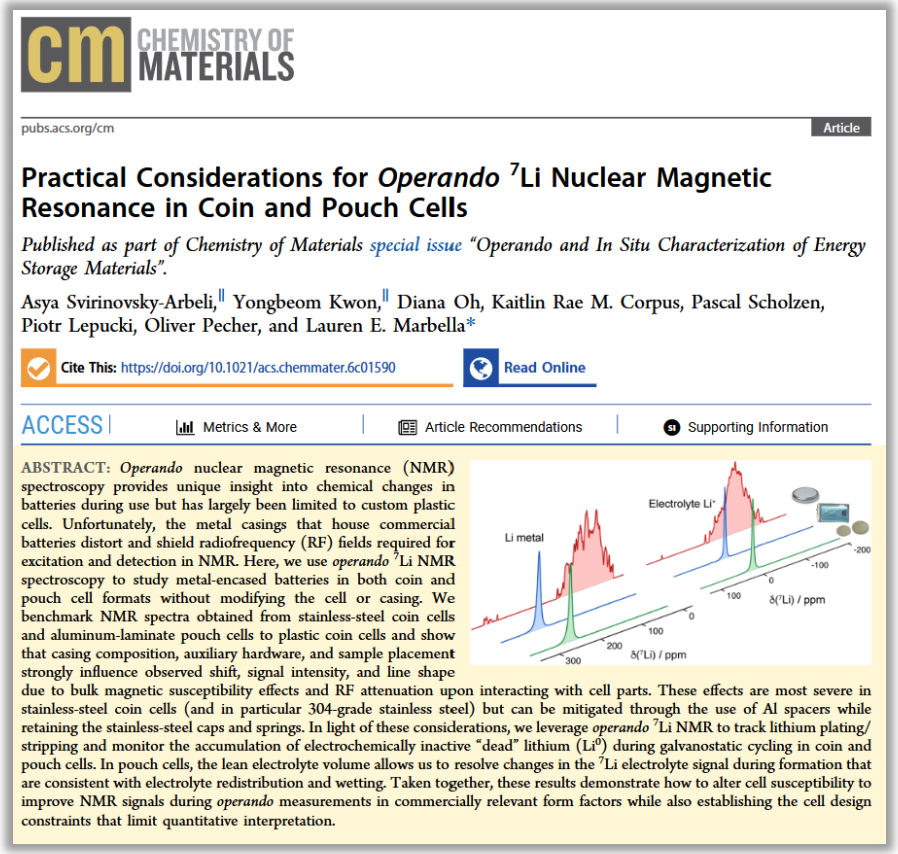
Operando NMR on coin and pouch cells

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CHEMISTRY OF MATERIALS

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Practical Considerations for *Operando* ^{7}Li Nuclear Magnetic Resonance in Coin and Pouch Cells

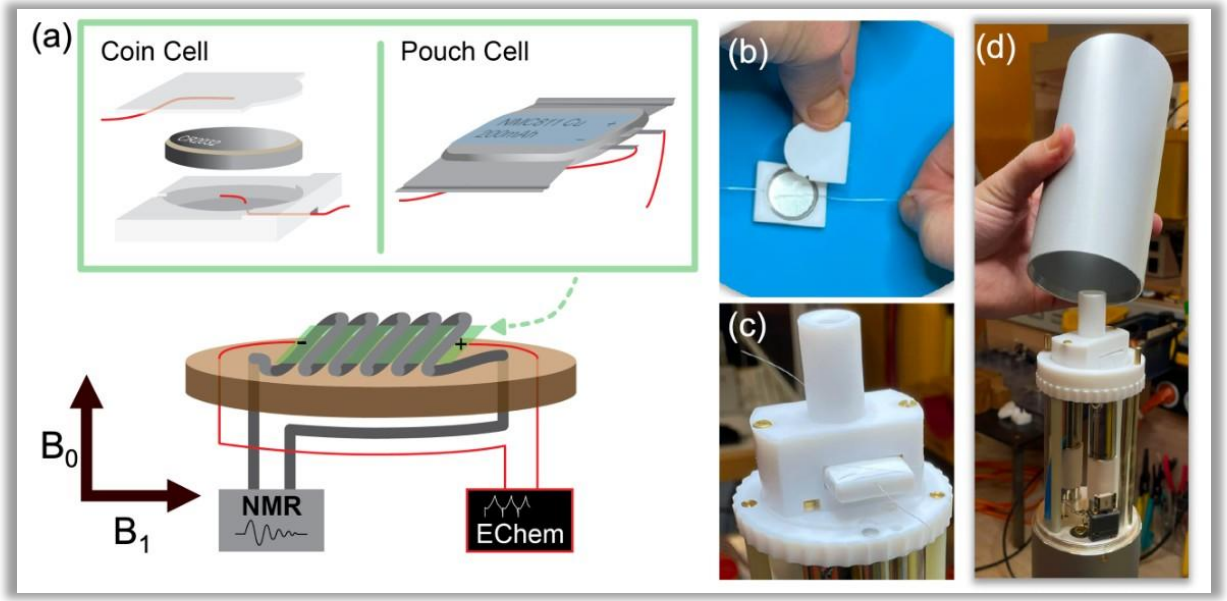
Published as part of Chemistry of Materials special issue "Operando and In Situ Characterization of Energy Storage Materials".

Asya Svirinovsky-Arbeli,‖ Yongbeom Kwon,‖ Diana Oh, Kaitlin Rae M. Corpus, Pascal Scholzen, Piotr Lepucki, Oliver Pecher, and Lauren E. Marbella*

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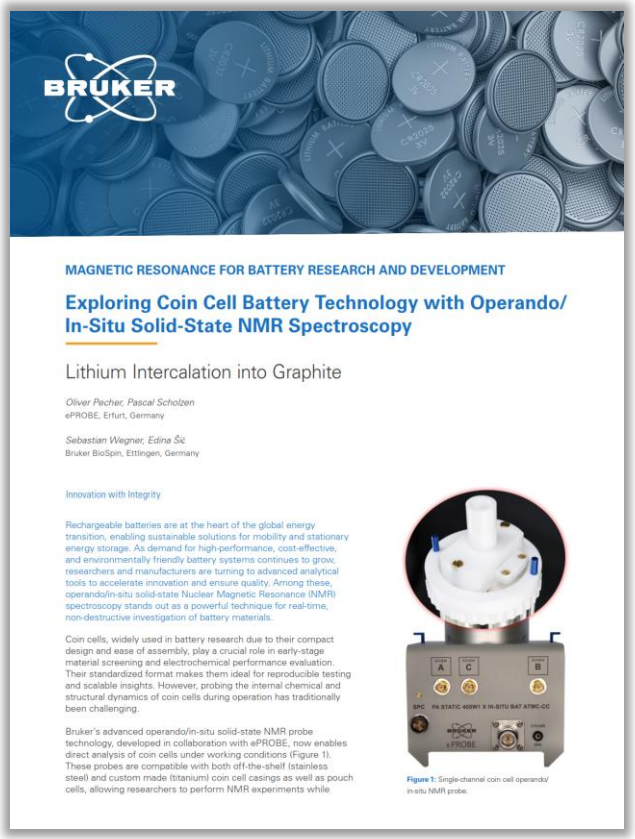
ABSTRACT: *Operando* nuclear magnetic resonance (NMR) spectroscopy provides unique insight into chemical changes in batteries during use but has largely been limited to custom plastic cells. Unfortunately, the metal casings that house commercial batteries distort and shield radiofrequency (RF) fields required for excitation and detection in NMR. Here, we use *operando* ^{7}Li NMR spectroscopy to study metal-encased batteries in both coin and pouch cell formats without modifying the cell or casing. We benchmark NMR spectra obtained from stainless-steel coin cells and aluminum-laminate pouch cells to plastic coin cells and show that casing composition, auxiliary hardware, and sample placement strongly influence observed shift, signal intensity, and line shape due to bulk magnetic susceptibility effects and RF attenuation upon interacting with cell parts. These effects are most severe in stainless-steel coin cells (and in particular 304-grade stainless steel) but can be mitigated through the use of Al spacers while retaining the stainless-steel caps and springs. In light of these considerations, we leverage *operando* ^{7}Li NMR to track lithium plating/stripping and monitor the accumulation of electrochemically inactive "dead" lithium (Li^{0}) during galvanostatic cycling in coin and pouch cells. In pouch cells, the lean electrolyte volume allows us to resolve changes in the ^{7}Li electrolyte signal during formation that are consistent with electrolyte redistribution and wetting. Taken together, these results demonstrate how to alter cell susceptibility to improve NMR signals during *operando* measurements in commercially relevant form factors while also establishing the cell design constraints that limit quantitative interpretation.

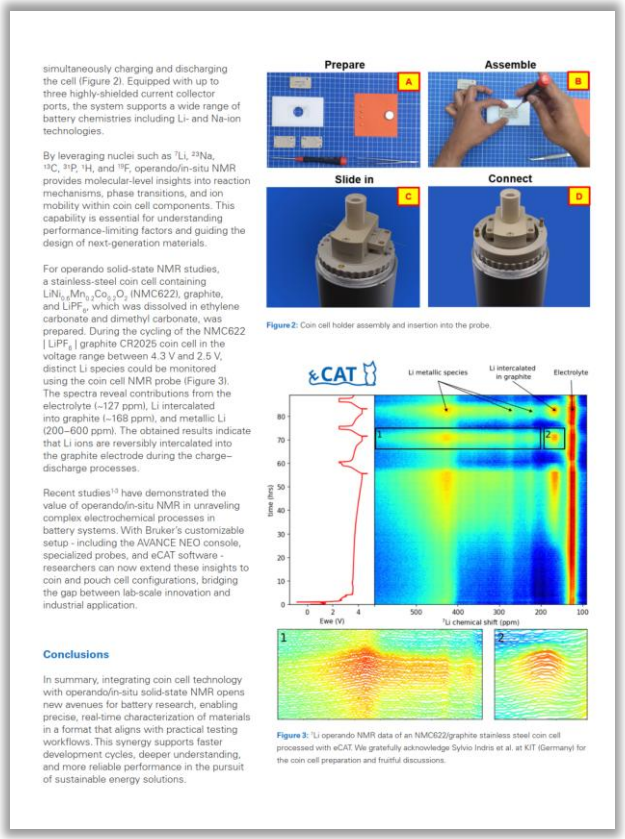


https://doi.org/10.1021/acs.chemmater.6c01590

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https://www.bruker.com/en/resources/library/application-notes-mr/coin-cell-battery.html

Operando NMR on coin and pouch cells – Probe designs

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Single-channel probe broadband ^{13}C to ^{31}P range incl. smart probe control



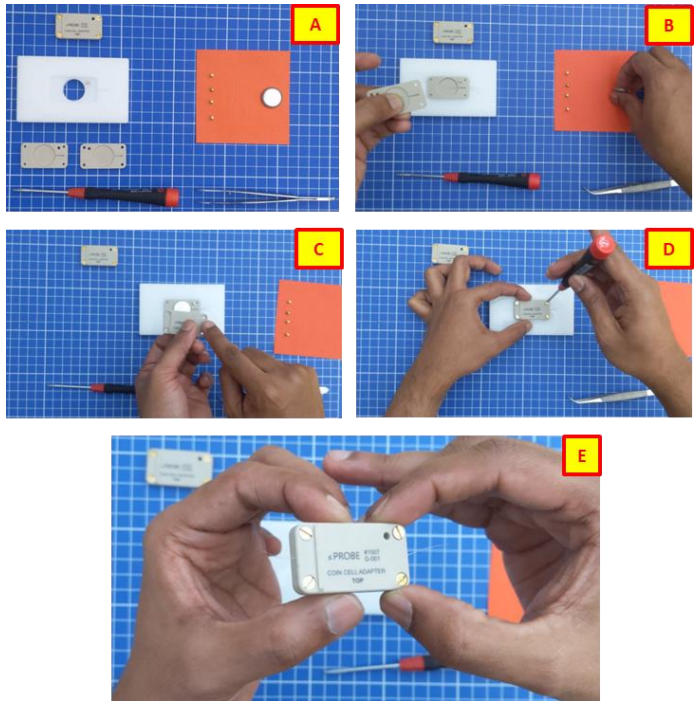
Double-channel probe with fixed frequencies, e.g. ^{19}F and ^{7}Li

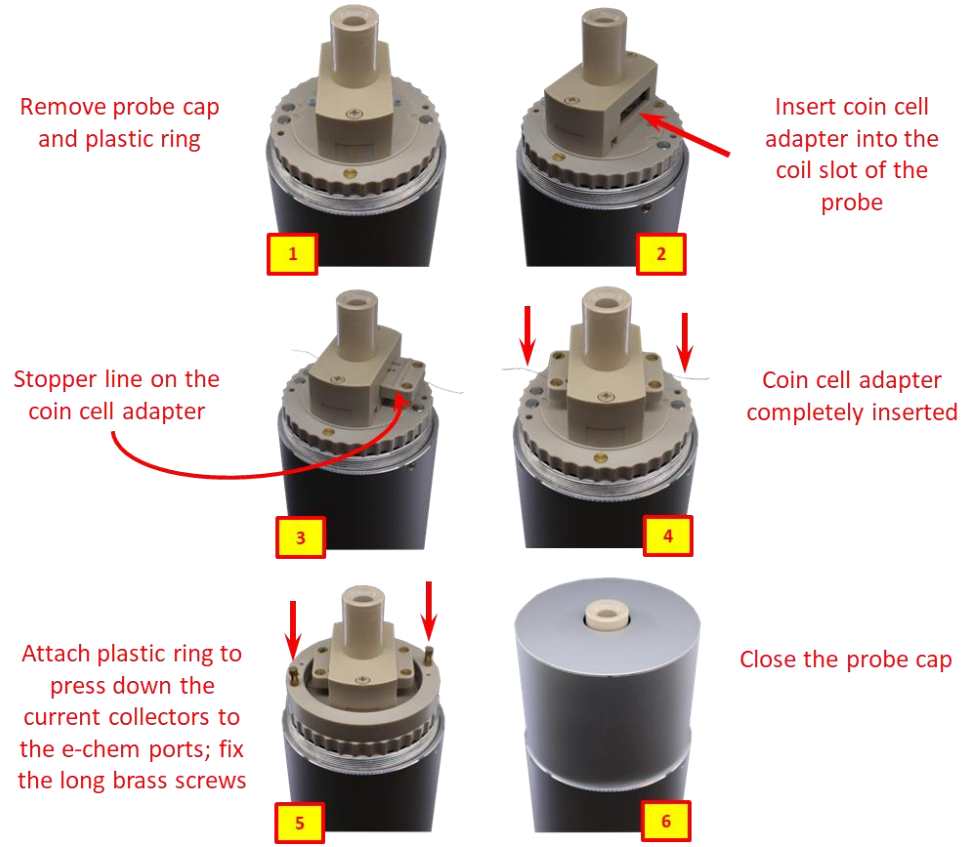


https://eprobe.tech/probes/probes-coin-cell

Operando NMR on coin and pouch cells – Handling







https://eprobe.tech/probes/probes-coin-cell

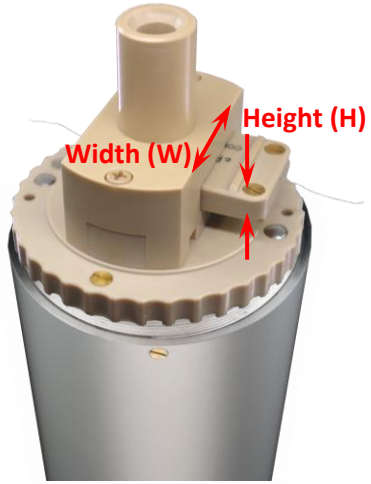
Operando NMR on coin and pouch cells – Dimensions

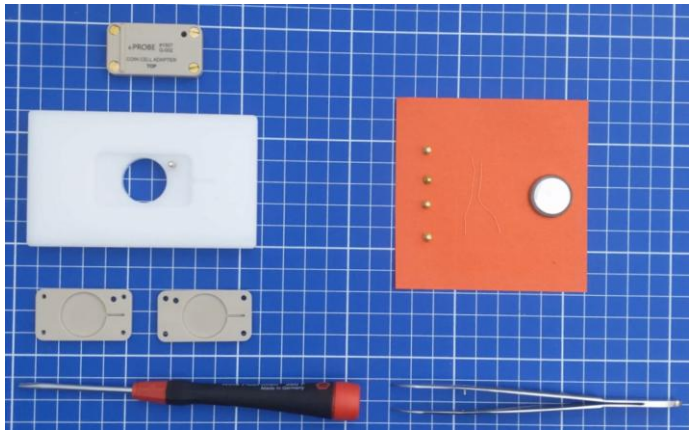
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Coil/sample slot
Width x Height
24 x 7 mm

Coin cell adapter
for coin cells
up to size CR2032

Pouch cells(1)
Length x Width x Height
26 x 24 x 7 mm





Exemplary picture only.



https://eprobe.tech/probes/probes-coin-cell
(1) Pouch cells up to 37 mm in length fit inside the probe sample slot but part of the pouch cell will then be outside of the coil.

Operando NMR on coin cells – Application

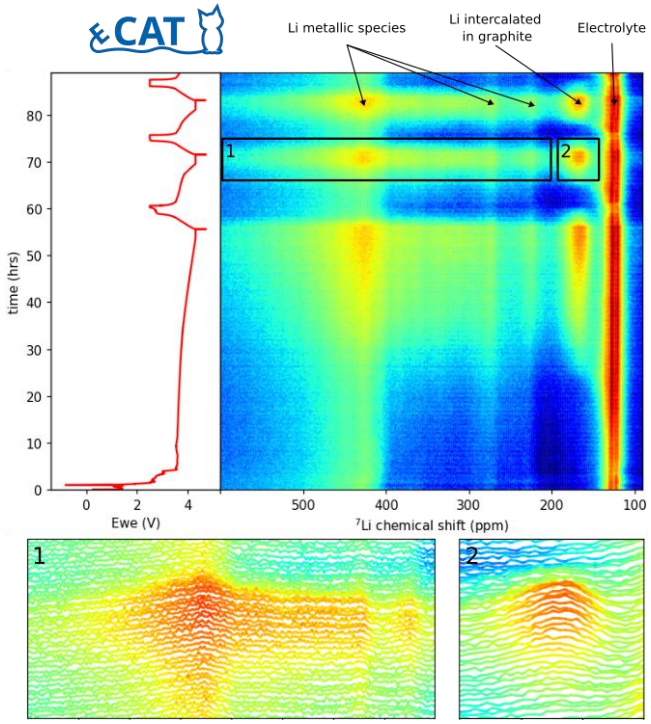
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Steel coin cell:

NMC | Graphite full-cell:

→ Detection of metallic Li and intercalated species

→ NMR signal shifts, but electrolyte as reliable reference

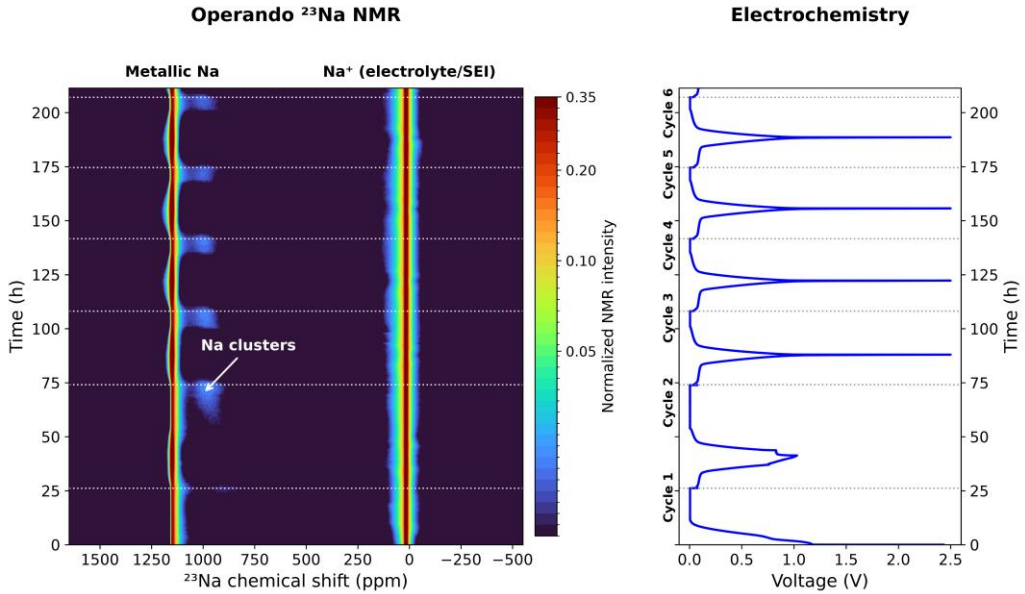


Titanium coin cell:

Na metal | Berliner Schwarz® half-cell:

→ Same cell preparation and cycling performance as steel coin cells

→ Signal shifts and line broadening comparable to plastic cell capsules



BBL BERLIN BATTERY LAB

BAM Bundesanstalt für Materialforschung und -prüfung

Experiments and data analysis performed by Nahom Asres, BAM Battery NMR Lab

https://www.bruker.com/en/resources/library/application-notes-mr/coin-cell-battery.html

https://bam.de/en/topics/projects/solid-state-and-operando-nmr-analytics-for-battery-research