Journal of Magnetic Resonance 306 (2019) 91–97

Contents lists available at ScienceDirect

Journal of Magnetic Resonance

journal homepage: www.elsevier.com/locate/jmr

Materializing opportunities for NMR of solids

Bradley F. Chmelka

Department of Chemical Engineering, University of California, Santa Barbara, CA 93106, USA

ARTICLE INFO

Article history:
Received 4 June 2019
Revised 20 July 2019
Accepted 20 July 2019
Available online 22 July 2019

ABSTRACT

Advancements in sensitivity and resolution of NMR of solids are opening a bonanza of fundamental and technological opportunities in materials science. Many of these are at the boundaries of related disciplines that provide creative inputs to motivate the development of new methodologies and possibilities for new applications. As Boltzmann limitations are surmounted by dynamic-nuclear-polarization- and laser-enhanced hyperpolarization techniques, the correlative benefits of multidimensional NMR are becoming more and more impactful. Nevertheless, there are limits, and the atomic-level information provided by solid-state NMR will be most useful in combination with state-of-the-art diffraction, microscopy, computational, and materials synthesis methods. Collectively these can be expected to lead to design criteria that will promote discovery of new materials, lead to novel or improved material properties, catalyze new applications, and motivate further methodological advancements.

© 2019 Elsevier Inc. All rights reserved.

1. Introduction

A crystallographer, an electron microscopist, and a NMR spectroscopist were visiting Barcelona and admiring Casa Batlló (Fig. 1), a famous building designed by the renowned architect Antoni Gaudí that appears to be have been flash-frozen from a complex fluid mixture into stone. The crystallographer pondered the edifice for some time at a distance with a furrowed brow, before pronouncing that because the structure lacked long-range periodicity, it could not be classified, would not be stable, and probably should not be called a "building." In the meantime, the electron microscopist had approached the exterior of Casa Batlló and got as close as possible to examine it at several points with the aid of a magnifying glass, before announcing with satisfaction that the sample had nice recognizable patterns that were clearly a mixture of crystalline components, though to establish how they were related to each other would require a full statistical analysis and research project to fund a two-year study. The solid-state NMR spectroscopist thought, "those blokes are sure scattered," and while examining the intricacies of the exterior up close, exclaimed with wonder about the rich commingling of crystalline and disordered components, the boundaries between them, their different textures, and "such magnificent detail!", but got carried away and didn't comment on the building itself.

Gaudí's buildings and architectural innovations were revolutionary because of their flowing designs, heterogeneous compositions, and the engineering means by which they were constructed and stabilized. Though they differ in scale and function from today's pursuits in materials science, there are many analogies between the disparate disciplines. These include quests to control complicated distributions of material compositions and structures over multiple length and time scales to achieve properties that would not be possible otherwise. In designing his buildings, Gaudí assembled different materials components in clever ways to achieve a combination of structural and visual properties and function. By analogy, materials scientists and chemical engineers seek to combine different components, typically at an atomic level, to obtain combinations of a tremendously diverse range of properties, including mechanical, catalytic, photophysical, electrochemical, rheological, separation, and other applications. Furthermore, the number of possible atomic compositions, local configurations, and long-range structures and distributions are vast and depend on synthesis and processing conditions. Establishing how these attributes collectively influence macroscopic material properties is a central objective of modern materials science and to which insights from magnetic resonance can crucially contribute.

Recent and looming advancements in solid-state NMR provide exciting new opportunities to understand the properties of heterogeneous materials at an atomic level. Such properties notably include the local compositions, structures, and dynamics of multicomponent solids and, if undertaken in combination with scattering analyses, how local characteristics are correlated with elemental distributions, extents of crystallinity, larger-scale structures, their evolution over time, and their influences on technologically important properties. In pursuing such insights, recent NMR

E-mail address: BRADC@ENGINEERING.UCSB.EDU

https://doi.org/10.1016/j.jmr.2019.07.051
1090-7807/© 2019 Elsevier Inc. All rights reserved.

Fig. 1. Casa Batlló, a UNESCO World Heritage site in Barcelona designed in 1904 by the renowned Catalan architect Antoni Gaudí. Photo credit: Xavier Salvatella.

methodology advancements and development goals that target enhanced signal sensitivity and resolution provide exciting new capabilities and prospects for probing and answering previously intractable materials questions. Many of the other articles in this issue of *JMR* discuss methodology objectives and visions that directly intersect with those presented below, some of which will be highlighted for their specific relevance to understanding complicated heterogeneous solids and their properties.

2. "Oh, don't be so sensitive..."

... are words that NMR spectroscopists have never been known to utter (at least not in scientific contexts.) This is especially the case for those studying materials in which dilute or low-gyromagnetic-ratio (γ) nuclei or surfaces are of principal interest. Among the most exciting developments of the past 10 years has been the demonstration of the extraordinary utility of hyperpolarization methods to enhance NMR signal intensities. These notably include dynamic nuclear polarization (DNP) to enhance the NMR signals of sparse surface or near-surface species, investigations of which have been largely beyond the capabilities of X-ray and electron scattering methods or limited mostly to NMR investigations of materials with high surface areas or extensive interfacial contacts. Solid-state DNP-NMR methods circumvent several of these limitations and are paving the way for diverse current efforts to enhance NMR signal sensitivity and guiding future progress.

The current and envisioned development of new DNP-NMR capabilities for solids are vibrant and the expansion of the opportunities that they present to materials science are exciting. These derive from a combination of relatively recent factors, notably the ground-breaking advancements in hardware by Griffin and coworkers [1,2], the availability of reliable commercial DNP-NMR instrumentation and radical species, the development and adaptation of polarization transfer methodologies for solids by the Lyon and EPFL [3,4] groups and others. In particular, applications of surface-enhanced DNP studies to solid materials have benefited crucially from the design and syntheses of new stable organic radical species [5,6]. Whereas dissolution DNP-NMR investigations of proteins or other biomolecules have tended to converge on similar formulations of biradicals in partially deuterated glycerol-water mixtures, applications of DNP-NMR to solids cover a much broader range of formulations, according to the hydrophilicity, hydrophobicity, reactivity, solubility and available area of the solid surfaces present. Different biradical architectures, including asymmetric molecules [6], are being developed and tested for their compatibilities with different solvents, materials, and conditions, all of which must be selected judiciously to achieve high DNP-NMR signal enhancements. Partnerships among DNP-NMR groups, organic chemists, materials scientists, and commercial vendors have been crucial and synergistic factors to the burgeoning use of DNP-NMR for investigations of increasingly diverse materials. These have, for example, enabled measurements of nuclei with low natural abundances and/or low-γ values, such as ^{13}C and ^{29}Si in mesoporous solids [3,4], ^{17}O in inorganic oxides [7,8], ^{15}N in pharmaceutical particles [9], ^{43}Ca in biomineral hydroxyapatites [10], ^{89}Y in solid-state proton conductors [11], and ^{113}Cd in nanoparticles [12], to be analyzed without the need for isotopic enrichment. This versatile sensitivity windfall has enabled previously infeasible analyses of near-surface species [13–16] without the need for expensive or impractical isotopic enrichment or dilute adsorbed species on low-surface-area solids [17,18], including acquisition of multidimensional NMR spectra. This has opened exciting new prospects for measuring and understanding technologically important heterogeneous solids, such as catalysts, structural materials, semiconductors, pharmaceuticals, etc.

Nevertheless, though a great deal of progress has been made, the scope of new candidate materials, compositional and structural questions, types of nuclei, and measurement conditions remains huge. These present new methodological opportunities for NMR at the interfaces among solid-state chemistry, physics, materials science, and engineering, many of which are driven by motivating materials applications. For example, demonstration that endogenous radicals can yield high DNP-NMR signal enhancements [19–22] opens opportunities for characterizing paramagnetic materials without the need to use exogenous radical-solvent formulations that can affect sample surface properties and complicate the analyses. Further progress in this area would enable DNP-NMR studies of bulk materials containing paramagnetic centers, such as carbonaceous solids, e.g., porous carbons and conjugated polymers, doped or defect-containing semiconductors and phosphors, and cementious structural solids, including those with paramagnetic transition-metal species. A severe limitation of current methods of solid-state DNP-NMR has been the low temperatures (<100 K) that are typically required. This is due to the need for conditions that promote long spin-lattice relaxation times, electron-nuclear hyperfine interactions, and nuclear dipole-dipole couplings to transfer spin polarization from the radical moieties to nuclear spins on the solid surface of interest. Achieving high levels of spin polarization at higher temperatures [23,24] represents an important quest that will substantially extend the types of solid systems that can be analyzed by NMR under more chemically, biologically, or industrially relevant conditions. In combination with recent advancements in DNP-NMR at very high fields (>19 Tesla) and under fast MAS (>60 kHz) conditions, these will benefit structural investigations of quadrupolar nuclei or dilute dopant species by increasing signal sensitivity and spectral resolution that could only be dreamed of a decade ago. Such anticipated progress should lead to greatly expanded applications in industry, where it will be possible by analyze materials after use to examine the effects of process conditions or ageing, including forensic investigations after process upsets or material failures.

3. Some benefits of being resolutionally-challenged

However, even if high spin polarizations can be achieved, resolving NMR signals spectrally, spatially, or temporally often presents extensive challenges though also opportunities. The vast majority of technologically important materials are heterogeneous in their compositions, structures, or dynamics, which inherently lead to distributions of the local atomic environments to which nuclear spins are sensitive. In solids, these frequently result in broadened NMR signals that complicate analyses, but which also contain extensive information on the interactions that account for the broadening and which can be managed by judicious selection of experimental conditions. For example, the combination of ever-higher magnetic fields, faster-MAS probeheads, and advances in pulse-sequence design and implementation reduce the difficulties presented by homogeneously broadened signals, such as those arising from dipolar, second-order quadrupolar, or large chemical shift anisotropy interactions. These enable high-resolution solid-state NMR spectra to be acquired and powerful analyses to be conducted on bulk materials with strong dipole-dipole interactions, such as small organic molecules [25,26] and polymers [27,28] or heterogeneous catalysts [29] containing high concentrations of ^{1}H or ^{19}F. This is similarly the case for glassy or crystalline inorganic solids with non-integer spin >½ quadrupolar nuclei, such as ^{17}O, ^{27}Al, ^{73}Ge, and others that exhibit large quadrupolar coupling constants, in particular those with high atomic masses [30–33]. For other materials containing nuclei with large chemical shift or Knight shift anisotropies (e.g., ^{195}Pt in catalysts [34,35] and ^{207}Pb [36,37]) or large nuclear quadrupole moments (e.g., ^{14}N and ^{35}Cl in pharmaceutical compounds [9], ^{59}Co [38] and ^{105}Pd [39] in catalysts, ^{139}La in ferroelectrics [40], or ^{115}In in semiconductors [41]), megahertz-broad spectra can be acquired by ultra-wideline NMR techniques [42–44]. Additional challenges and opportunities arise when paramagnetic species are present that displace NMR signals [45], which can be used to resolve signals associated with atomic environments near the paramagnetic centers that are responsible for crucial functional properties. These notably include technologically important solid-state materials for battery applications [46,47], solid-state lighting [48], catalysts [49], and glasses [50], which may similarly benefit from higher magnetic fields and faster MAS conditions.

Independent of the availability of high magnetic fields or fast MAS probeheads, the diverse menu of spin interactions that operate on NMR-active nuclei, whether singly or in coupled ensembles, provide a vast scope of opportunities to enhance spectral resolution. Multinuclear, and especially multidimensional, experiments enable an enormous range of atomic-level material properties to be selectively probed, guided by the insights sought on local compositions, structures, and dynamics, even for inhomogeneously broadened NMR spectra. These include the uses of isotropic and anisotropic chemical shift interactions to characterize the nearby chemical environments of nuclei, *J* interactions between coupled spin pairs to establish covalent bonding of atoms, dipolar interactions to probe through-space proximities of coupled spin pairs, quadrupolar interactions to assess the local electric field gradients and symmetry near nuclei, Knight shifts or paramagnetic displacements to probe the respective influences of electron conductivity or unpaired electron spins near nuclei. Importantly, most such interactions can be correlated in two (and occasionally more) dimensions to enhance, often dramatically, the spectral resolution and associated information content. For example, challenging materials where correlation NMR techniques have recently been used to obtain novel insights include the uses of 2D ^{1}H{^{1}H} double-quantum spectra to analyze the local structures and dynamics of polymer chains [51,52], ^{13}C spectral-editing to identify functional groups and their distributions in soils, lignins, and carbonaceous fuels [53,54], 2D ^{13}C{^{1}H} dipolar-mediated spectra to identify surface sites in organometallic-functionalized heterogeneous catalysts [55], 1D ^{13}C{^{27}Al} rotational-echo saturation-pulse double-resonance measurements of adsorbed organic molecules on supported Pd-γ-alumina [56], 2D ^{29}Si{^{29}Si} or ^{29}Si{^{31}P} *J*-mediated spectra to correlate distributions of atomic connectivities and bond angles in silica-based glasses [57,58], 2D ^{27}Al{^{29}Si} *J*-mediated spectra to determine the distributions of Al atoms among framework sites in an aluminosilicate zeolite catalyst [59], 2D ^{31}P{^{27}Al} *J*-mediated spectra to distinguish Al-P atom pairs in aluminophosphate frameworks [60], and 2D ^{71}Ga{^{15}N} spectra to correlate ^{71}Ga and ^{15}N Knight shifts in GaN nanoparticles [61]. The intensities and positions of the correlated signals generally depend on the strengths of the respective nuclear spin interactions, which themselves depend on intrinsic material compositions and structures of interest, such as the types and number of coupled nuclei and their separation distances. Correlated signal intensities also depend on experimental conditions, such as temperature and pressure which influence nanoscale mobilities, macroscopic sample reorientation trajectories and rates, or pulse sequences. Nevertheless, for a given material and set of conditions, correlated intensities in multidimensional NMR spectra enable the positions and the lineshapes of the signals to be resolved and confidently established. These in turn can be subsequently used to deconvolute quantitative direct-excitation 1D NMR spectra to confidently estimate the relative populations of atomic species in a heterogeneous material. The influences of the above interactions and processes can be selectively probed in different combinations, among different nuclei, and under different conditions, as motivated by the insights sought on local material composition, structure, dynamics, and associated properties.

Enhanced signal sensitivity and resolution also provide improved prospects for obtaining spatially-resolved spectra or images of heterogeneous solids. These tend to be significantly more challenging to acquire for solids, compared to conventional magnetic resonance imaging (MRI) of soft matter. This is because spin-spin relaxation times (T_2) must be sufficiently long (>0.1 ms) and adequate spectral resolution can be difficult to achieve in the presence of magnetic field gradients that are typically used to encode spatial positions within a sample. For example, under MAS conditions, rotating magnetic-field gradients [62–64] or stray-field gradients [65,66] have been used to encode positions based on field-dependent differences in spin-relaxation times or local spin interactions within rotating solid samples to establish distributions of atomic environments on length scales of order 100 μm. Similar spatial resolution can also be obtained in non-rotating samples to probe ^{1}H-containing material compositions and structures near a surface (to a depth of ca. 25 mm) by using stray fields from a portable permanent magnet assembly [67]. This has enabled non-invasive analyses of cultural heritage materials, such as for the restoration and conservation of paintings, sculptures, or carvings, the curing or aging of polymers, coatings or weathered surfaces of structural materials, as well as biosolids [68]. Another surface-imaging approach combines aspects of scanning probe microscopy with laser-enhanced polarization techniques to achieve spatial resolution < 1 μm for ^{1}H-containing surface structures by exploiting local dipolar fields near a hyperpolarized semiconductor micro-tip [69]. Whereas the large majority of such imaging applications have till now relied on abundant nuclei, such as ^{1}H, ^{19}F, ^{31}P, or isotopic enrichment, it is exciting to contemplate new opportunities that would arise by combining imaging techniques with new reconstruction protocols and hyperpolarization methods to detect and map dilute or low-γ nuclei with high spatial resolution. Advancing capabilities to image or acquire spatially resolved NMR spectra near the surfaces of intact monolithic solids have great diagnostic and forensic potential for

analyses of culturally, historically, or economically valuable materials.

Among the most powerful aspects of NMR is its utility for monitoring dynamic phenomena in heterogeneous solids. Spin-relaxation times, NMR lineshapes, exchange spectra, or diffusion-mediated intensities are sensitive to dynamical processes that can influence NMR signals over an enormous range of time scales that span at least nine orders of magnitude. Many of these are well-established methods that similarly benefit from the enhancements of signal sensitivity and spectral resolution discussed above. The dynamics of molecules, for example, are central to understanding the mechanical and rheological behaviors of polymers [51,52], inorganic glasses and melts [70], colloidal suspensions [71], as well as the diffusion of adsorbed molecules in porous solids [72,73], ion mobility in bulk solids [74,75], and transitions between phases [37,76]. Variable-temperature analyses feature prominently in such investigations, as do multinuclear techniques that probe and correlate the local environments and interactions of different component nuclei, providing a wealth of complementary information.

One important area for materials research in which there has been significant recent progress is the use of *in situ* or *operando* NMR techniques to monitor chemical reaction processes. These methods typically involve the integration of a chemical reactor (e.g., a catalyst bed) [77–82] or an electrochemical device, such as a battery or supercapacitor electrode [83–90], within a probehead that enables temperature, pressure, and/or voltage to be controlled and reactants, products, or electric current to flow into and out of the probehead, while NMR spectra (e.g., ^{1}H, ^{6}Li, ^{7}Li, ^{23}Na, ^{31}P, ^{51}V, etc.) are being acquired. For solids, the resolution of signals from distinct species requires that they be sufficiently separated due to differences in their intrinsic shifts, often under static conditions where molecular motion or exchange averages anisotropic interactions or by sample reorientation (e.g., MAS). Analyses can be conducted under steady-state conditions or transiently to monitor chemical or physical changes to a system over time; the latter requires sufficient sensitivity such that the acquisitions of signal-averaged spectra are fast relative to the transient processes of interest. New opportunities can be envisioned for *in situ* NMR analyses of solid-state materials at non-ambient pressures, during simultaneous optical excitation, or for radioactive species. Such *in situ* analyses present attractive opportunities to measure the properties of NMR-active nuclei in materials under actual process or operating conditions, enabling detailed atomic-level insights to be correlated directly with macroscopic catalytic, electrochemical, or other types of kinetic properties in real time.

4. Aiming high

New innovations and advancements push the frontiers of the fields to which they can be applied, and vice-versa. With respect to solid-state NMR, vibrant and continuous innovation in engineering and integration of ever-higher-field superconducting magnets, higher-power microwave or other polarization sources, and ultra-fast magic-angle-spinning (MAS) or *in situ* probeheads will open new opportunities for materials science. Increased sensitivity and resolution of NMR signals will benefit the study of heterogeneous solids in positive and relatively evolutionary ways through the availability of uniform magnetic fields that exceed 30 Tesla [91,92] (with intense on-going efforts to integrate high-T_c superconductor technology into new magnet designs) and ever faster MAS probeheads with spinning rates above 120 kHz. These hardware improvements, however, are requiring more time and investment to achieve, with development costs and purchase prices increasing accordingly. As a consequence, fewer such systems will likely be produced and may be concentrated in center-like facilities, where their accessibility will be shared. This would have several advantages, including dispersed benefits from the large investments required and access to local technical specialists. However, state-of-the-art "conventional" spectrometer instrumentation will still need to be widely available to screen samples and to optimize measurement conditions to justify and efficiently use the specialized facilities.

But one never has enough signal sensitivity. All of the above advancements and progress notwithstanding, a holy grail of sensitivity enhancement remains the development of generalized means to optically excite or detect NMR signals. There has been notable progress in using lasers to enhance dramatically nuclear spin polarization in gases, which has also been shown to be transferrable under certain conditions to NMR-active nuclei in heterogeneous liquids or at solid surfaces [93]. This includes NMR studies that rely on laser-polarized noble gases or hyperpolarized *para*-hydrogen to monitor transient reaction processes in heterogeneous fluid-solid systems [94,95]. Progress has also been made in using laser light or microwaves to excite nuclear spins in opto-electronic materials [24,96–99], including under conditions of fast field cycling by high-speed sample shuttling [100], or at terahertz frequencies [101] to conduct high-field EPR analyses. These advancements are enabling hyperpolarized NMR investigations of materials under increasingly diverse ranges of magnetic field strengths, irradiation conditions, and temperatures. Being able to combine the high sensitivity of optical detection (perhaps even single photons) with the high spectral resolution and spin-interaction selectivity of NMR will open new horizons and applications in materials science and more broadly.

Looking to the future, applications of solid-state NMR spectroscopy in materials science are at the cusp of rapid expansion. Diverse, powerful, and increasingly sensitive techniques yield atomic-level insights that are crucial for understanding and improving the properties of heterogeneous materials with complicated compositions and structures, including both ordered and disordered regions. The combination of high NMR spectral resolution, correlated spin interactions, and quantitativeness (though typically not in a single measurement) is unique among characterization methods and accounts for the versatile utility of solid-state NMR in materials science and allied disciplines. Nevertheless, NMR is not a panacea. Greater understanding and impact result when the atomic-scale information obtained from NMR is used in combination with scattering, microscopy, or other complementary techniques. In particular, integrating such multiscale characterization insights with first-principles modeling [102–109], advanced structure-search approaches [110,111], and machine learning protocols [112] will accelerate the analyses and enable the understanding to be generalized. The impacts will be amplified further by partnering with and providing feedback to materials synthesis and engineering groups to optimize materials properties and device or process performances. Beyond answering materials-specific questions, such interdisciplinary collaborations can be expected to motivate additional NMR methodological advancements, lead to design criteria that will spur materials discovery, and catalyze new applications.

Antoni Gaudí of course did not need NMR spectroscopy, electron microscopy, or X-ray diffraction to design and construct his iconic buildings. His genius was to apply innovative understanding of how dissimilar component materials could be integrated over multiple length scales to achieve novel macroscopic structural effects. Such an approach is analogous to the goals of contemporary (and undoubtedly future) materials scientists, which are now being extended down to atomic dimensions. Solid-state NMR will be vital for providing and correlating atomic-level data and properties to guide materials synthesis and processing

conditions, for use as inputs in machine learning analyses, and to aid predictive modeling and design of material properties. The future for NMR of solids is materially exciting with tremendous opportunities for impact, especially in combination with advancing states-of-the-art in related techniques and disciplines. Gaudí had no idea what he was missing.

Acknowledgements

I thank my coworkers, especially the graduate students and post-doctoral researchers in my research group over the years, and our many collaborating partners. These include in particular Prof. Lyndon Emsley at the Ecole Polytechnique Fédérale de Lausanne (EPFL), Switzerland, Dr. Anne Lesage and Dr. Guido Pintacuda at the Centre de RMN à Très Hauts Champs in Lyon, France, Prof. Alex Pines and Prof. Clayton Radke at the University of California, Berkeley, Prof. Hans Wolfgang Spiess at the Max-Planck-Institut für Polymerforschung in Mainz, Germany, and Prof. Ernest Giralt and his group at the Universitat de Barcelona. Prof. Niklas Hedin and Prof. Andrew Pell provided helpful comments during preparation of the manuscript. I am grateful for funding support from diverse agencies, including industrial partners who have invested in fundamental understanding of complicated engineering materials by solid-state NMR. Much of our work has been conducted using the Shared Facilities of the UCSB Materials Research Laboratory, which is supported by the MRSEC program of the U. S. National Science Foundation under Award No. DMR-1720256 and which is a member of the NSF-funded Materials Research Facilities Network (www.mrfn.org).

References

[1] T. Maly, G.T. Debelouchina, V.S. Bajaj, K.-N. Hu, C.-G. Joo, M.L. Mak-Jurkauskas, J.R. Sirigiri, P.C.A. van der Wel, J. Herzfeld, R.J. Temkin, R.G. Griffin, Dynamic nuclear polarization at high magnetic fields, J. Chem. Phys. 128 (2008) 052211.
[2] Q.Z. Ni, E. Daviso, T.V. Can, E. Markhasin, S.K. Jawla, T.M. Swager, R.J. Temkin, J. Herzfeld, R.G. Griffin, High frequency dynamic nuclear polarization, Acc. Chem. Res. 46 (2013) 1933–1941.
[3] A. Lesage, M. Lelli, D. Gajan, M.A. Caporini, V. Vitzthum, P. Miéville, J. Alauzun, A. Roussey, C. Thieuleux, A. Mehdi, G. Bodenhausen, C. Copéret, L. Emsley, Surface enhanced NMR spectroscopy by dynamic nuclear polarization, J. Am. Chem. Soc. 132 (2010) 15459–15461.
[4] M. Lelli, D. Gajan, A. Lesage, M.A. Caporini, V. Vitzthum, P. Miéville, F. Héroguel, F. Rascón, A. Roussey, C. Thieuleux, M. Boualleg, L. Veyre, G. Bodenhausen, C. Copéret, L. Emsley, Fast characterization of functionalized silica materials by silicon-29 surface-enhanced NMR spectroscopy using dynamic nuclear polarization, J. Am. Chem. Soc. 133 (2011) 2104–2107.
[5] A. Zagdoun, G. Casano, O. Ouari, G. Lapadula, A.J. Rossini, M. Lelli, M. Baffert, D. Gajan, L. Veyre, W.E. Maas, M. Rosay, R.T. Weber, C. Thieuleux, C. Copéret, A. Lesage, P. Tordo, L. Emsley, A slowly relaxing rigid biradical for efficient dynamic nuclear polarization surface-enhanced NMR spectroscopy: expeditious characterization of functional group manipulation in hybrid materials, J. Am. Chem. Soc. 134 (2012) 2284–2291.
[6] D. Wisser, G. Karthikeyan, A. Lund, G. Casano, H. Karoui, M. Yulikov, G. Menzildjian, A.C. Pinon, A. Purea, F. Engelke, S.R. Chaudhari, D. Kubicki, A.J. Rossini, I.B. Moroz, D. Gajan, C. Copéret, G. Jeschke, M. Lelli, L. Emsley, A. Lesage, O. Ouari, BDPA-nitroxide biradicals tailored for efficient dynamic nuclear polarization enhanced solid-state NMR at magnetic fields up to 21.1 T, J. Am. Chem. Soc. 140 (2018) 13340–13349.
[7] F. Blanc, L. Sperrin, D.A. Jefferson, S. Pawsey, M. Rosay, C.P. Grey, Dynamic nuclear polarization enhanced natural abundance ^{17}O spectroscopy, J. Am. Chem. Soc. 135 (2013) 2975–2978.
[8] N.J. Brownbill, D. Gajan, A. Lesage, L. Emsley, F. Blanc, Oxygen-17 dynamic nuclear polarisation enhanced solid-state NMR spectroscopy at 18.8 T, Chem. Commun. 53 (2017) 2563–2566.
[9] S.L. Veinberg, K.E. Johnston, M.J. Jaroszewicz, B.M. Kispal, C.R. Mireault, T. Kobayashi, M. Pruski, R.W. Schurko, Natural abundance ^{14}N and ^{15}N solid-state NMR of pharmaceuticals and their polymorphs, Phys. Chem. Chem. Phys. 18 (2016) 17713–17730.
[10] D. Lee, C. Leroy, C. Crevant, L. Bonhomme-Coury, F. Babonneau, D. Laurencin, C. Bonhomme, G. De Paëpe, Interfacial Ca^{2+} environments in nanocrystalline apatites revealed by dynamic nuclear polarization enhanced ^{43}Ca NMR spectroscopy, Nat. Commun. 8 (2017) 14104.
[11] F. Blanc, L. Sperrin, D. Lee, R. Dervişoğlu, Y. Yamazaki, S.M. Haile, G. De Paëpe, C.P. Grey, Dynamic nuclear polarization NMR of low-γ nuclei: structural insights into hydrated yttrium-doped $BaZrO_3$, J. Phys. Chem. Lett. 5 (2014) 2431–2436.
[12] L. Piveteau, T.-C. Ong, B.J. Walder, D.N. Dirin, D. Moscheni, B. Schneider, J. Bär, L. Protesescu, N. Masciocchi, A. Guagliardi, L. Emsley, C. Copéret, M.V. Kovalenko, Resolving the core and the surface of CdSe quantum dots and nanoplatelets using dynamic nuclear polarization enhanced PASS–PIETA NMR spectroscopy, ACS Cent. Sci. 4 (2018) 1113–1125.
[13] A.C. Pinon, U. Skantze, J. Viger-Gravel, S. Schantz, L. Emsley, Core–shell structure of organic crystalline nanoparticles determined by relayed dynamic nuclear polarization NMR, J. Phys. Chem. A 122 (2018) 8802–8807.
[14] S. Björgvinsdóttir, B.J. Walder, A.C. Pinon, L. Emsley, Bulk nuclear hyperpolarization of inorganic solids by relay from the surface, J. Am. Chem. Soc. 140 (2018) 7946–7951.
[15] L. Zhao, A.C. Pinon, L. Emsley, A.J. Rossini, DNP-enhanced solid-state NMR spectroscopy of active pharmaceutical ingredients, Magn. Reson. Chem. 56 (2018) 583–609.
[16] D. Xiao, S. Xu, N.J. Brownbill, S. Paul, L.-H. Chen, S. Pawsey, F. Aussenac, B.-L. Su, X. Han, X. Bao, Z. Liu, F. Blanc, Fast detection and structural identification of carbocations on zeolites by dynamic nuclear polarization enhanced solid-state NMR, Chem. Sci. 9 (2018) 8184–8193.
[17] R.P. Sangodkar, B.J. Smith, D. Gajan, A.J. Rossini, L.R. Roberts, G.P. Funkhouser, A. Lesage, L. Emsley, B.F. Chmelka, Influences of dilute organic adsorbates on the hydration of low-surface-area silicates, J. Am. Chem. Soc. 137 (2015) 8096–8112.
[18] B.J. Walder, C. Berk, W.-C. Liao, A.J. Rossini, M. Schwarzwälder, U. Pradere, J. Hall, A. Lesage, C. Copéret, L. Emsley, One- and two-dimensional high-resolution NMR from flat surfaces, ACS Cent. Sci. 5 (2019) 515–523.
[19] R.A. Wind, F.E. Anthonio, M.J. Duijvestijn, J. Smidt, J. Trommel, G.M.C. de Vette, Experimental setup for enhanced ^{13}C NMR spectroscopy in solids using dynamic nuclear polarization, J. Magn. Reson. 52 (1983) 424–434.
[20] H. Jayakumar, S. Dhomkar, J. Henshaw, C.A. Meriles, Spin readout via spin-to-charge conversion in bulk diamond nitrogen-vacancy ensembles, Appl. Phys. Lett. 113 (2018) 122404.
[21] T. Wolf, S. Kumar, H. Singh, T. Chakrabarty, F. Aussenac, A.I. Frenkel, D.T. Major, M. Leskes, Endogenous dynamic nuclear polarization for natural abundance ^{17}O and lithium NMR in the bulk of inorganic solids, J. Am. Chem. Soc. 141 (2019) 451–462.
[22] E. Verde-Sesto, N. Goujon, H. Sardon, P. Ruiz, T.V. Huynh, F. Elizalde, D. Mecerreyes, M. Forsyth, L.A. O'Dell, DNP NMR studies of crystalline polymer domains by copolymerization with nitroxide radical monomers, Macromolecules 51 (2018) 8046–8053.
[23] A. Zagdoun, G. Casano, O. Ouari, M. Schwarzwälder, A.J. Rossini, F. Aussenac, M. Yulikov, G. Jeschke, C. Copéret, A. Lesage, P. Tordo, L. Emsley, Large molecular weight nitroxide biradicals providing efficient dynamic nuclear polarization at temperatures up to 200 K, J. Am. Chem. Soc. 135 (2013) 12790–12797.
[24] P.R. Zangara, J. Henshaw, D. Pagliero, A. Ajoy, J.A. Reimer, A. Pines, C.A. Meriles, Two-electron-spin ratchets as a platform for microwave-free dynamic-nuclear-polarization of arbitrary material targets, Nano Lett. 19 (2019) 2389–2396.
[25] T. Kobayashi, K. Mao, P. Paluch, A. Nowak-Król, J. Sniechowska, Y. Nishiyama, D.T. Gryko, M.J. Potrzebowski, M. Pruski, Study of intermolecular interactions in the corrole matrix by solid-state NMR under 100 kHz MAS and theoretical calculations, Angew. Chem. Int. Ed. 52 (2013) 14108–14111.
[26] Y. Nishiyama, Fast magic-angle sample spinning solid-state NMR at 60–100 kHz for natural abundance samples, Solid-State NMR 78 (2016) 24–36.
[27] G.N.M. Reddy, M. Malon, A. Marsh, Y. Nishiyama, S.P. Brown, Fast magic-angle spinning three-dimensional NMR experiment for simultaneously probing H-H and N-H proximities in solids, Anal. Chem. 88 (2016) 11412–11419.
[28] R. Zhang, K.H. Mroue, A. Ramamoorthy, Proton-based ultrafast magic angle spinning solid-state NMR spectroscopy, Acc. Chem. Res. 50 (2017) 1105–1113.
[29] A.A. Malär, S. Dong, G. Kehr, G. Erker, B.H. Meier, T. Wiegand, Characterization of H_2-splitting products of frustrated Lewis pairs: Benefit of fast magic-angle spinning, ChemPhysChem 20 (2019) 672–679.
[30] M. Haouas, J. Trébosc, C. Roch-Marchal, E. Cadot, F. Taulelle, C. Martineau-Corcos, High-field ^{95}Mo and ^{183}W static and MAS NMR study of polyoxometalates, Magn. Reson. Chem. 55 (2017) 902–908.
[31] N.M. Trease, T.M. Clark, P.J. Grandinetti, J.F. Stebbins, S. Sen, Bond length-bond angle correlation in densified silica—Results from ^{17}O NMR spectroscopy, J. Chem. Phys. 146 (2017) 184505.
[32] V.K. Michaelis, P.M. Aguiar, V.V. Terskikh, S. Kroeker, Germanium-73 NMR of amorphous and crystalline GeO_2, Chem. Commun. (2009) 4660–4662.
[33] C. Leroy, D.L. Bryce, Recent advances in solid-state nuclear magnetic resonance spectroscopy of exotic nuclei, Prog. NMR Spectr. 109 (2018) 160–199.
[34] M. Soorholtz, L.C. Jones, D. Samuelis, C. Weidenthaler, R.J. White, M.-M. Titirici, D.A. Cullen, T. Zimmermann, M. Antonietti, J. Maier, R. Palkovits, B.F. Chmelka, F. Schüth, Local platinum environments in a solid analogue of the molecular Periana catalyst, ACS Catal. 6 (2016) 2332–2340.
[35] T. Kobayashi, F.A. Perras, T.W. Goh, T.L. Metz, W. Huang, M. Pruski, DNP-enhanced ultrawideline solid-state NMR spectroscopy: studies of platinum in metal-organic frameworks, J. Phys. Chem. Lett. 7 (2016) 2322–2327.
[36] T. Kobayashi, F.A. Perras, A. Murphy, Y. Yao, J. Catalano, S.A. Centeno, C. Dybowski, N. Zumbulyadis, M. Pruski, DNP-enhanced ultrawideline ^{207}Pb

solid-state NMR spectroscopy: an application to cultural heritage science, Dalton Trans. 46 (2017) 3535–3540.
[37] C.E. Avalos, B.J. Walder, J. Viger-Gravel, A. Magrez, L. Emsley, Chemical exchange at the ferroelectric phase transition of lead germanate revealed by solid state ^{207}Pb nuclear magnetic resonance, Phys. Chem. Chem. Phys. 21 (2019) 1100–1109.
[38] K.M.N. Burgess, C.M. Widdifield, Y. Xu, C. Leroy, D.L. Bryce, Structural insights from ^{59}Co solid-state NMR experiments on organocobalt(I) catalysts, ChemPhysChem 19 (2018) 227–236.
[39] T.J.N. Hooper, T.A. Partridge, G.J. Rees, D.S. Keeble, N.A. Powell, M.E. Smith, I.P. Mikheenko, L.E. Macaskie, P.T. Bishop, J.V. Hanna, Direct solid state NMR observation of the ^{105}Pd nucleus in inorganic compounds and palladium metal systems, Phys. Chem. Chem. Phys. 20 (2018) 26734–26743.
[40] A.L. Paterson, U. Werner-Zwanziger, J.W. Zwanziger, Network connectivity and crystallization in the transparent ferroelectric nanocomposite $LaBGeO_5$, J. Phys. Chem. C 123 (2019) 11860–11873.
[41] S.-H. Cho, S. Ghosh, Z.J. Berkson, J.A. Hachtel, J. Shi, X. Zhao, L.C. Reimnitz, C.J. Dahlman, Y. Ho, A. Yang, Y. Liu, J.C. Idrobo, B.F. Chmelka, D. Milliron, Syntheses of colloidal F:In_2O_3 cubes: Fluorine-induced faceting and infrared plasmonic responses, Chem. Mater. 31 (2019) 2661–2676.
[42] R. Schurko, Ultra-wideline solid-state NMR spectroscopy, Acc. Chem. Res. 46 (2013) 1985–1995.
[43] P. Paluch, A.G.M. Rankin, J. Trébosc, O. Lafon, J.-P. Amoureux, Analysis of HMQC experiments applied to a spin ½ nucleus subject to very large CSA, Solid State NMR 100 (2019) 11–25.
[44] S. Wi, R.W. Schurko, L. Frydman, Broadband adiabatic inversion cross-polarization phenomena in the NMR of rotating solids, Solid-State NMR 94 (2018) 31–53.
[45] A.J. Pell, G. Pintacuda, C.P. Grey, Paramagnetic NMR in solution and the solid state, Prog. NMR Spectr. 111 (2019) 1–271.
[46] C. Grey, N. Dupré, NMR studies of cathode materials for lithium-ion rechargeable batteries, Chem. Rev. 104 (2004) 4493–4512.
[47] R.J. Messinger, M. Ménétrier, E. Salager, A. Boulineau, M. Duttine, D. Carlier, J.-M. Ateba Mba, L. Croguennec, C. Masquelier, D. Massiot, M. Deschamps, Revealing defects in crystalline lithium-ion battery electrodes by solid-state NMR: applications to $LiVPO_4F$, Chem. Mater. 27 (2015) 5212–5221.
[48] N.C. George, J. Brgoch, A.J. Pell, C. Cozzan, A. Jaffe, G. Dantelle, A. Llobet, G. Pintacuda, R. Seshadri, B.F. Chmelka, Correlating local compositions and structures with macroscopic optical properties of Ce^{3+}-doped $CaSc_2O_4$, and efficient green-emitting phosphor, Chem. Mater. 29 (2017) 3538–3546.
[49] H.T. Kreissl, M.M.J. Li, Y.-K. Peng, K. Nakagawa, T.J.N. Hooper, J.V. Hanna, A. Shepherd, T.-S. Wu, Y.-L. Soo, S.C.E. Tsang, Structural studies of bulk to nanosize niobium oxides with correlation to their acidity, J. Am. Chem. Soc. 139 (2017) 12670–12680.
[50] J.H. Baltisberger, P. Florian, E.G. Keeler, P.A. Phyo, K.J. Sanders, P.J. Grandinetti, Modifier cation effects on ^{29}Si nuclear shielding anisotropies in silicate glasses, J. Magn. Reson. 268 (2016) 95–106.
[51] H.W. Spiess, 50th anniversary perspective: the importance of NMR spectroscopy to macromolecular science, Macromolecules 50 (2017) 1761–1777.
[52] A. Mordvinkin, M. Suckow, F. Böhme, R.H. Colby, C. Creton, K. Saalwächter, Hierarchical sticker and sticky chain dynamics in self-healing butyl rubber ionomers, Macromolecules 52 (2019) 4169–4184.
[53] J. Mao, X. Cao, D.C. Olk, W. Chu, K. Schmidt-Rohr, Advanced solid-state NMR spectroscopy of natural organic matter, Prog. NMR Spectr. 100 (2017) 17–51.
[54] W. Chu, X. Cao, K. Schmidt-Rohr, J.E. Birdwell, J. Mao, Investigation into the effect of heteroatom content on kerogen structure using advanced ^{13}C solid-state nuclear magnetic resonance spectroscopy, Energy Fuels 33 (2019) 645–653.
[55] C. Copéret, W.-C. Liao, C.P. Gordon, T.-C. Ong, Active sites in supported single-site catalysts: an NMR perspective, J. Am. Chem. Soc. 139 (2017) 10588–10596.
[56] F.A. Perras, J.D. Padmos, R.L. Johnson, L.-L. Wang, T.J. Schwartz, T. Kobayashi, J. H. Horton, J.A. Dumesic, B.H. Shanks, D.D. Johnson, M. Pruski, Characterizing substrate–surface interactions on alumina-supported metal catalysts by dynamic nuclear polarization-enhanced double-resonance NMR spectroscopy, J. Am. Chem. Soc. 139 (2017) 2702–2709.
[57] D.J. Srivastava, J.H. Baltisberger, P. Florian, F. Fayon, R.A. Shakhovoy, M. Deschamps, N. Sadiki, P.J. Grandinetti, Correlating structural distributions in silica glass with two-dimensional *J*-resolved spectroscopy, Phys. Rev. B 98 (2018) 134202.
[58] J. Ren, H. Eckert, Superstructural units involving six-coordinated silicon in sodium phosphosilicate glasses detected by solid-state NMR spectroscopy, J. Phys. Chem. C 122 (2018) 27620–27630.
[59] Z.J. Berkson, M.-F. Hsieh, S. Smeets, D. Gajan, A. Lund, A. Lesage, D. Xie, S.I. Zones, L.B. McCusker, C. Baerlocher, B.F. Chmelka, Preferential siting of Al heteroatoms in the zeolite catalyst Al-SSZ-70, Angew. Chem. Intl. Ed. 58 (2019) 6255–6259.
[60] Q. Wang, J. Trébosc, Y. Li, O. Lafon, S. Xin, J. Xu, B. Hu, N. Feng, J.-P. Amoureux, F. Deng, Uniform signal enhancement in MAS NMR of half-integer quadrupolar nuclei using quadruple-frequency sweeps, J. Magn. Reson. 293 (2018) 92–103.
[61] J.P. Yesinowski, Z.J. Berkson, S. Cadars, A.P. Purdy, B.F. Chmelka, Metallic character and correlated distributions of electronic structure in bulk and nanocrystalline GaN established by MAS-NMR, Phys. Rev. B 95 (2017) 235201.
[62] D.G. Cory, A.M. Reichwein, J.W.M. Van Os, W.S. Veeman, NMR images of rigid solids, Chem. Phys. Lett. 143 (1988) 467–470.
[63] U. Scheler, G. Schauss, B. Blümich, H.W. Spiess, Spatially resolved solid-state MAS-NMR-spectroscopy, Solid State NMR 6 (1996) 375–388.
[64] M. Yon, V. Sarou-Kanian, U. Scheler, J.-M. Bouler, B. Bujoli, D. Massiot, F. Fayon, Solid-state ^{31}P and ^{1}H chemical MR micro-imaging of hard tissues and biomaterials with magic angle spinning at very high magnetic field, Sci. Rep. 7 (2017) 8224.
[65] A. Wong, D. Sakellariou, Contrast STRAFI–MAS imaging, J. Magn. Reson. 206 (2010) 264–268.
[66] F.A. Perras, T. Kobayashi, M. Pruski, Magnetic resonance imaging of DNP enhancements in a rotor spinning at the magic angle, J. Magn. Reson. 264 (2016) 125–130.
[67] C. Rehorn, B. Blümich, Cultural heritage studies with mobile NMR, Angew. Chem. Int. Ed. 57 (2018) 7304–7312.
[68] E. Danieli, B. Blümich, Single-sided magnetic resonance profiling in biological and materials science, J. Magn. Reson. 229 (2013) 142–154.
[69] C.A. Meriles, Optically detected nuclear magnetic resonance at the sub-micron scale, J. Magn. Reson. 176 (2005) 207–214.
[70] P. Florian, A. Novikov, J.W.E. Drewitt, L. Hennet, V. Sarou-Kanian, D. Massiot, H.E. Fischer, D.R. Neuville, Structure and dynamics of high-temperature strontium aluminosilicate melts, Phys. Chem. Chem. Phys. 20 (2018) 27865–27877.
[71] E.O. Fridjonsson, J.D. Seymour, Colloid particle transport in a microcapillary: NMR study of particle and suspending fluid dynamics, Chem. Eng. Sci. 153 (2016) 165–173.
[72] D.E. Favre, D.J. Schaefer, S.M. Auerbach, B.F. Chmelka, Direct measurement of intercage hopping in strongly adsorbing guest-zeolite systems, Phys. Rev. Lett. 81 (1998) 5852–5855.
[73] V.J. Witherspoon, J. Xu, J.A. Reimer, Solid-state NMR investigations of carbon dioxide gas in metal–organic frameworks: Insights into molecular motion and adsorptive behavior, Chem. Rev. 118 (2018) 10033–10048.
[74] J. Langer, D.L. Smiley, A.D. Bain, G.R. Goward, M. Wilkening, An unexpected pathway: ^{6}Li-exchange NMR spectroscopy points to vacancy-driven out-of-plane Li-ion hopping in crystalline Li_2SnO_3, J. Phys. Chem. C 120 (2016) 3130–3138.
[75] S. Breuer, B. Stanje, V. Pregartner, S. Lunghammer, I. Hanzu, M. Wilkening, Fluorine translational anion dynamics in nanocrystalline ceramics: SrF_2-YF_3 solid solutions, Crystals 8 (2018) 122.
[76] M.T. Seifrid, G.N.M. Reddy, C. Zhou, B.F. Chmelka, G.C. Bazan, Direct observation of the relationship between molecular topology and bulk morphology for a π-conjugated material, J. Am. Chem. Soc. 141 (2019) 5078–5082.
[77] X.L. Wang, W. Liu, Y.-Y. Yu, Y. Song, W.Q. Fang, D. Wei, X.-Q. Gong, Y.-F. Yao, H.G. Yang, Operando NMR spectroscopic analysis of proton transfer in heterogeneous photocatalytic reactions, Nat. Commun. 7 (2016) 11918.
[78] S.A. Morris, G.P.M. Bignami, Y. Tian, M. Navarro, D.S. Firth, J. Čejka, P.S. Wheatley, D.M. Dawson, W.A. Slawinski, D.S. Wragg, R.E. Morris, S.E. Ashbrook, In situ solid-state NMR and XRD studies of the ADOR process and the unusual structure of zeolite IPC-6, Nat. Chem. 9 (2017) 1012–1018.
[79] L. Qi, R. Alamillo, W.A. Elliott, A. Andersen, D.W. Hoyt, E.D. Walter, K.S. Han, N. M. Washton, R.M. Rioux, J.A. Dumesic, S.L. Scott, Operando solid-state NMR observation of solvent-mediated adsorption-reaction of carbohydrates in zeolites, ACS Catal. 7 (2017) 3489–3500.
[80] A.J. Oosthoek-de Vries, P.J. Nieuwland, J. Bart, K. Koch, J.W.G. Janssen, P.J.M. van Bentum, F.P.J.T. Rutjes, H.J.G.E. Gardeniers, A.P.M. Kentgens, Inline reaction monitoring of amine-catalyzed acetylation of benzyl alcohol using a microfluidic stripline nuclear magnetic resonance setup, J. Am. Chem. Soc. 141 (2019) 5369–5380.
[81] M. Leutzsch, A.J. Sederman, L.F. Gladden, M.D. Mantle, In situ reaction monitoring in heterogeneous catalysts by a benchtop NMR spectrometer, Magn Reson. Imag. 56 (2019) 138–143.
[82] X. Zhou, C. Wang, Y. Chu, J. Xu, Q. Wang, G. Qi, X. Zhao, N. Feng, F. Deng, Observation of an oxonium ion intermediate in ethanol dehydration to ethene on zeolite, Nat. Commun. 10 (2019) 1961.
[83] R. Bhattacharyya, B. Key, H. Chen, A.S. Best, A.F. Hollenkamp, C.P. Grey, In situ NMR observation of the formation of metallic lithium microstructures in lithium batteries, Nat. Mater. 9 (2010) 504–510.
[84] M. Tang, V. Sarou-Kanian, P. Melin, J.-B. Leriche, M. Ménétrier, J.-M. Tarascon, M. Deschamps, E. Salager, Following lithiation fronts in paramagnetic electrodes with in situ magnetic resonance spectroscopic imaging, Nat. Commun. 7 (2016) 13284.
[85] O. Pecher, J. Carretero-González, K.J. Griffith, C.P. Grey, Materials' methods: NMR in battery research, Chem. Mater. 29 (2017) 213–242.
[86] A.C. Forse, J.M. Griffin, C. Merlet, J. Carretero-Gonzalez, A.-R.O. Raji, N.M. Trease, C.P. Grey, Direct observation of ion dynamics in supercapacitor electrodes using in situ diffusion NMR spectroscopy, Nat. Energy 2 (2017) 16216.
[87] S.A. Krachkovskiy, J.M. Foster, J.D. Bazak, B.J. Balcom, G.R. Goward, Operando mapping of Li concentration profiles and phase transformations in graphite electrodes by magnetic resonance imaging and nuclear magnetic resonance spectroscopy, J. Phys. Chem. C 122 (2018) 21784–21791.
[88] J.L. Lorie Lopez, P.J. Grandinetti, A.C. Co, Phase transformations and capacity fade mechanism in Li_xSn nanoparticle electrodes revealed by operando ^{7}Li NMR, J. Mater. Chem. A 7 (2019) 10781–10794.

[89] X. Feng, M. Tang, S. O'Neill, Y.-Y. Hu, In situ synthesis and in operando NMR studies of a high-performance Ni_5P_4-nanosheet anode, J. Mater. Chem. A 6 (2018) 22240–22247.
[90] C.M.S. Lobo, B.F. Gomes, H. Bouzouma, E. Danieli, B. Blümich, L.A. Colnago, Improving in operando low field NMR copper electrodeposition analyses using inductively coupled coils, Electrochim. Acta 298 (2019) 844–851.
[91] E.G. Keeler, V.K. Michaelis, M.T. Colvin, I. Hung, P.L. Gor'kov, T.A. Cross, Z. Gan, R.G. Griffin, ^{17}O MAS NMR correlation spectroscopy at high magnetic fields, J. Am. Chem. Soc. 139 (2017) 17953–17963.
[92] C. Bonhomme, X. Wang, I. Hung, Z. Gan, C. Gervais, C. Sassoye, J. Rimsza, J. Du, M.E. Smith, J.V. Hanna, S. Sarda, P. Gras, C. Combes, D. Laurencin, Pushing the limits of sensitivity and resolution for natural abundance ^{43}Ca NMR using ultra-high magnetic field (35.2 T), Chem. Commun. 54 (2018) 9591–9594.
[93] H.C. Gaede, Y.-Q. Song, R.E. Taylor, E.J. Munson, J.A. Reimer, A. Pines, High-field cross polarization NMR from laser-polarized xenon to surface nuclei, Appl. Magn. Reson. 8 (1995) 373–384.
[94] E.W. Zhao, H. Zheng, K. Ludden, Y. Xin, H.E. Hagelin-Weaver, C.R. Bowers, Strong metal−support interactions enhance the pairwise selectivity of parahydrogen addition over Ir/TiO_2, ACS Catal. 6 (2016) 974–978.
[95] J.F.P. Colell, M. Emondts, A.W.J. Logan, K. Shen, J. Bae, R.V. Shchepin, G.X. Ortiz Jr., P. Spannring, Q. Wang, S.J. Malcolmson, E.Y. Chekmenev, M.C. Feiters, F.P.J. T. Rutjes, B. Blümich, T. Theis, W.S. Warren, Direct hyperpolarization of Nitrogen-15 in aqueous media with parahydrogen in reversible exchange, J. Am. Chem. Soc. 139 (2017) 7761–7767.
[96] R. Tycko, J.A. Reimer, Optical pumping in solid state nuclear magnetic resonance, J. Phys. Chem. 100 (1996) 13240–13250.
[97] M. Eickhoff, D. Suter, Pulsed optically detected NMR of single GaAs/AlGaAs quantum wells, J. Magn. Reson. 166 (2004) 69–75.
[98] A.K. Paravastu, S.E. Hayes, B.E. Schwickert, L.N. Dinh, M. Balooch, J.A. Reimer, Optical polarization of nuclear spins in GaAs, Phys. Rev. B. 69 (2004) 075203.
[99] M.M. Willmering, Z.L. Ma, M.A. Jenkins, J.F. Conley Jr., S.E. Hayes, Enhanced NMR with optical pumping yields ^{75}As signals selectively from a buried GaAs interface, J. Am. Chem. Soc. 139 (2017) 3930–3933.
[100] A. Ajoy, X. Lv, E. Druga, K. Liu, B. Safvati, A. Morabe, M. Fenton, R. Nazaryan, S. Patel, T.F. Sjolander, J.A. Reimer, D. Sakellariou, C.A. Meriles, A. Pines, Wide dynamic range magnetic field cycler: Harnessing quantum control at low and high fields, Rev. Sci. Instrum. 90 (2019) 013112.
[101] M.L.P. Bailey, A.T. Pierce, A.J. Simon, D.T. Edwards, G.J. Ramian, N.I. Agladze, M.S. Sherwin, Narrow-band water-based absorber with high return loss for terahertz spectroscopy, IEEE Trans. Terahertz Sci. Techn. 5 (2015) 961–966.
[102] D.H. Brouwer, S. Cadars, J. Eckert, Z. Liu, O. Terasaki, B.F. Chmelka, A general protocol for determining the structures of molecularly ordered but non-crystalline silicate frameworks, J. Am. Chem. Soc. 135 (2013) 5641–5655.
[103] D.S. Middlemiss, A.J. Ilott, R.J. Clément, F.C. Strobridge, C.P. Grey, Density functional theory-based bond pathway decompositions of hyperfine shifts: equipping solid-state NMR to characterize atomic environments in paramagnetic materials, Chem. Mater. 25 (2013) 1723–1734.
[104] C. Martineau, M. Allix, M.R. Suchomel, F. Porcher, F. Vivet, C. Legein, M. Body, D. Massiot, F. Taulelle, F. Fayon, Structure determination of Ba_5AlF_{13} by coupling electron, synchrotron and neutron powder diffraction, solid-state NMR and *ab initio* calculations, Dalton Trans. 45 (2016) 15565.
[105] F. Pourpoint, J. Yehl, M. Li, R. Gupta, J. Trébosc, O. Lafon, J.-P. Amoureux, T. Polenova, NMR crystallography of an oxovanadium(V) complex by an approach combining multinuclear magic angle spinning NMR, DFT, and spin dynamics simulations, ChemPhysChem 16 (2015) 1619–1626.
[106] S.E. Ashbrook, D. McKay, Combining solid-state NMR spectroscopy with first-principles calculations – a guide to NMR crystallography, Chem. Commun. 52 (2016) 7186–7204.
[107] J.M. Stratford, M. Mayo, P.K. Allan, O. Pecher, O.J. Borkiewicz, K.M. Wiaderek, K.W. Chapman, C.J. Pickard, A.J. Morris, C.P. Grey, Investigating sodium storage mechanisms in tin anodes: a combined pair distribution function analysis, density functional theory, and solid-state NMR approach, J. Am. Chem. Soc. 139 (2017) 7273–7286.
[108] A. Melnyk, M.J.N. Junk, M.D. McGehee, B.F. Chmelka, M.R. Hansen, D. Andrienko, Macroscopic structural compositions of π-conjugated polymers: combined insights from solid-state NMR and molecular dynamics simulations, J. Phys. Chem. Lett. 8 (2017) 4155–4160.
[109] A.S. Tatton, H. Blade, S.P. Brown, P. Hodgkinson, L.P. Hughes, S.O. Nilsson Lill, J.R. Yates, Improving confidence in crystal structure solutions using NMR crystallography: the case of β-piroxicam, Cryst. Growth Des. 18 (2018) 3339–3351.
[110] M. Zilka, D.V. Dudenko, C.E. Hughes, P.A. Williams, S. Sturniolo, W.T. Franks, C.J. Pickard, J.R. Yates, K.D.M. Harris, S.P. Brown, *Ab initio* random structure searching of organic molecular solids: assessment and validation against experimental data, Phys. Chem. Chem. Phys. 19 (2017) 25949–25960.
[111] D. McKay, R.F. Moran, D.M. Dawson, J.M. Griffin, S. Sturniolo, C.J. Pickard, A.J. Berry, S.E. Ashbrook, A picture of disorder in hydrous wadsleyite−under the combined microscope of solid-state NMR spectroscopy and *ab initio* random structure searching, J. Am. Chem. Soc. 141 (2019) 3024–3036.
[112] F.M. Paruzzo, A. Hofstetter, F. Musil, S. De, M. Ceriotti, L. Emsley, Chemical shifts in molecular solids by machine learning, Nat. Commun. 9 (2018) 4501.