

## GUEST EDITORIAL

# Frontiers in Solid-State NMR Technology

The development and understanding of solid materials is clearly one of the most dynamic driving forces in chemical sciences today. However, whether it be in biology or in materials science, there is a common bottleneck to further progress, leading to an overriding question being asked: "how can we determine atomic-level structure and dynamics of materials?" When single crystals are available, and if they are appropriate to the problem, then the answer may be straightforward. However, it is increasingly the case that materials may be inherently noncrystalline or that the process of obtaining a single crystal may denature the sample (e.g., glasses, heterogeneous catalysts, solar materials, batteries, pharmaceutical polymorphs, polymers, membrane proteins, protein fibrils, to name but a few). In such cases, there is a crying need for new methods of characterization.

Over the last 15 years or so, solid-state NMR has undergone a spectacular transition. It has now become a method of choice to determine structural features in systems ranging from paramagnetic battery materials and catalysts to amyloid fibrils and membrane proteins. In all these areas, solid-state NMR is now providing the structural answers that are allowing materials scientists and biologists to take the next steps to further developments, for longer lasting batteries or drug design, for example.

This transformation of the field has been driven by the introduction of a series of new techniques that allow the acquisition of more informative NMR spectra in a broader range of systems than was previously possible.

Spectral resolution and sensitivity have always been the key limiting factors for application of NMR in any field, but especially so in solids. The developments made have addressed these issues in order to improve resolution by faster and faster magic angle spinning, better heteronuclear or homonuclear spin decoupling, new sample preparation strategies, or creative isotopic labeling schemes. The introduction of sophisticated multidimensional NMR experiments has also dramatically improved resolution, either through methods that refocus anisotropic broadening or through multinuclear sequential correlations that spread

out and isolate resonances. These developments have been characterized by great diversity, with the result that today there are many tailor-made strategies that allow high-resolution spectra to be obtained in many different circumstances, from chemical shift distributions in glasses to detection of narrow proton resonances in proteins.

Of course, the increase of nearly a factor of two in available magnetic fields over the last 20 years has also made a marked contribution to both resolution and sensitivity of solid-state NMR.

Sensitivity improvements have also come from several other sources. The most obvious might be the widespread use of isotopic enrichment schemes, not only in biological samples but also increasingly in inorganic applications. The most successful studies today often involve a close partnership between synthetic chemists (or biologists) and spectroscopists, working together to adapt the sample in the best possible way to maximize the information that can be determined from the most appropriate NMR approach. Sensitivity has been improved considerably as the efficiency of multidimensional or multinuclear experiments has continuously increased, through the design of increasingly elegant or sophisticated magnetization transfer, recoupling, or decoupling schemes. A step change for solid-state NMR has been provided by the development of methods that allow *in situ* dynamic nuclear polarization to be coupled with low temperature (e.g., 100 K) magic-angle spinning systems. This brings about accelerations in acquisition times of more than 4 orders of magnitude, and the potential of this approach cannot be underestimated. It is already being developed and applied to the most challenging cases in both biological and materials systems.

This special issue, which was assembled together with Ivano Bertini, collects a comprehensive set of Accounts that reflect all these recent developments and their applications.

I cannot conclude without making special mention of Ivano. I think his arrival in the solid-state NMR field might have been one of the strongest indicators that the technique was maturing and beginning to play a useful role in

structural problems in proteins. Ivano was always on the lookout for new methods that could be put to use in solving structures or providing understanding of mechanisms in proteins (especially metalloproteins). He recognized the potential of solid-state NMR for paramagnetic proteins immediately once sample preparation protocols became available to obtain high-resolution spectra. I remember him declaring, with characteristic force, at a conference in Göttingen in 2006 that “the era of solid-state NMR is upon us.” By summer of last year, many of the projects in his laboratory, whether paramagnetic or not, involved solid-state NMR, and his group had become very active in developing new experimental approaches in the field.

In conclusion, I think these developments in Ivano's laboratory reflect well the ubiquitous nature of solid-state

NMR today, which has become one of the many tools that need to be applied, usually in concert, to solve modern problems of molecular characterization beyond the monocrystal! I hope the readers of this special issue will share this enthusiasm and be prompted to add solid-state NMR to their own repertoires, and I look forward to seeing where all this will lead.

Views expressed in this editorial are those of the authors and not necessarily the views of the ACS.

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