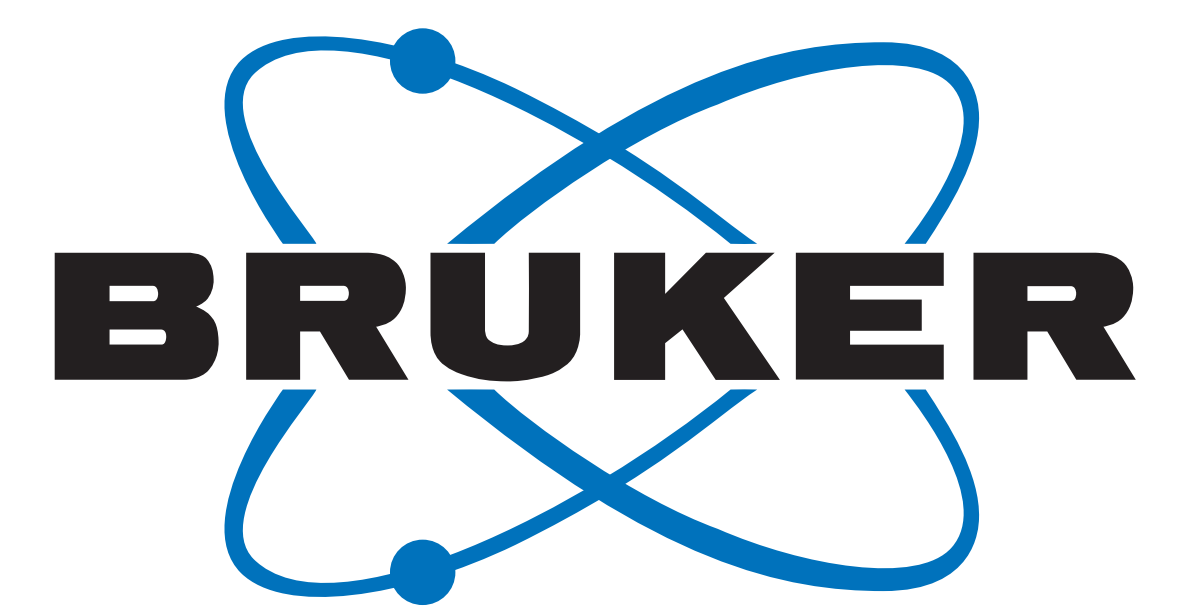


# CMP Broad-band iProbe: Maximum Versatility for MAS Experiments



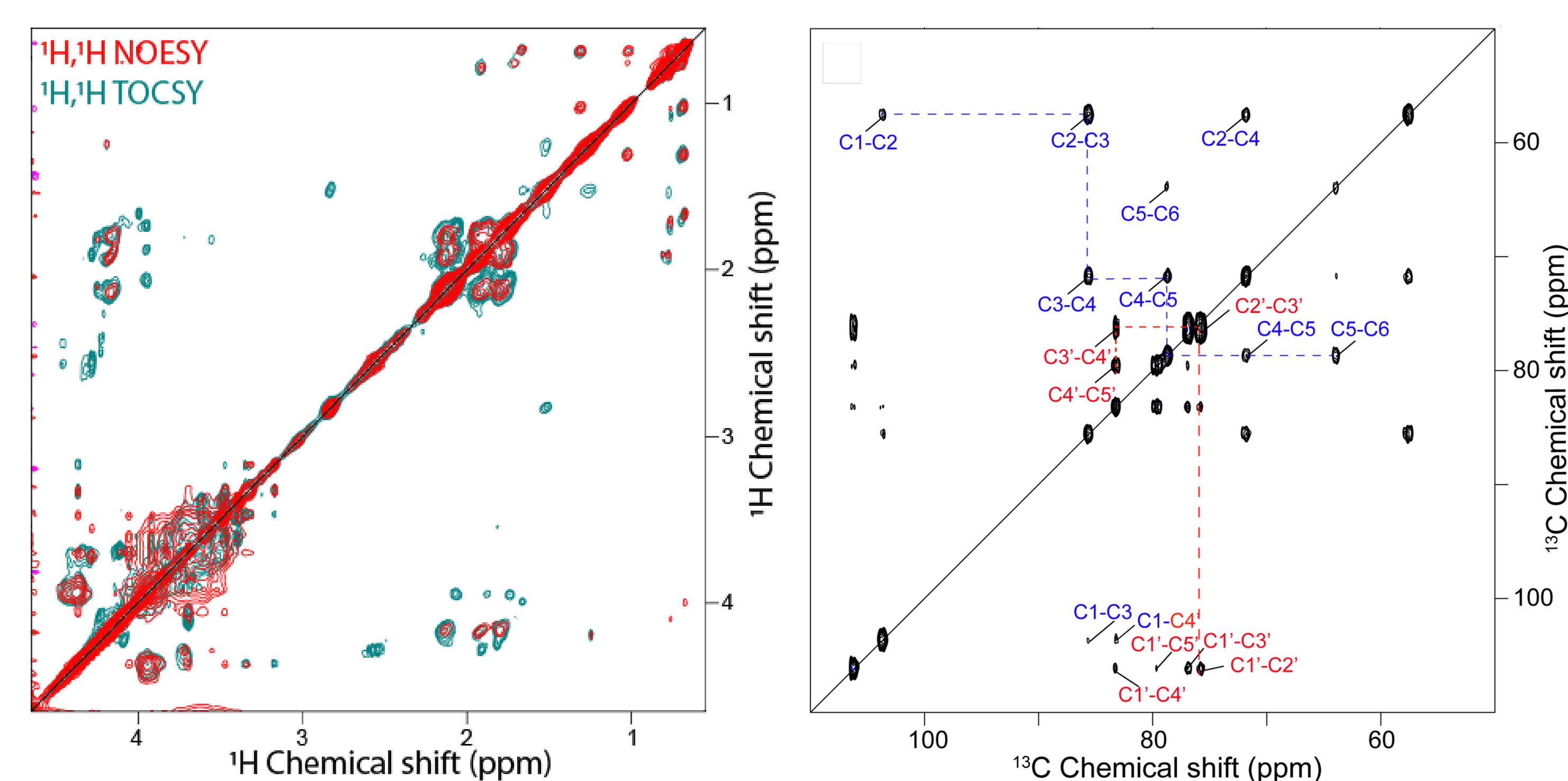
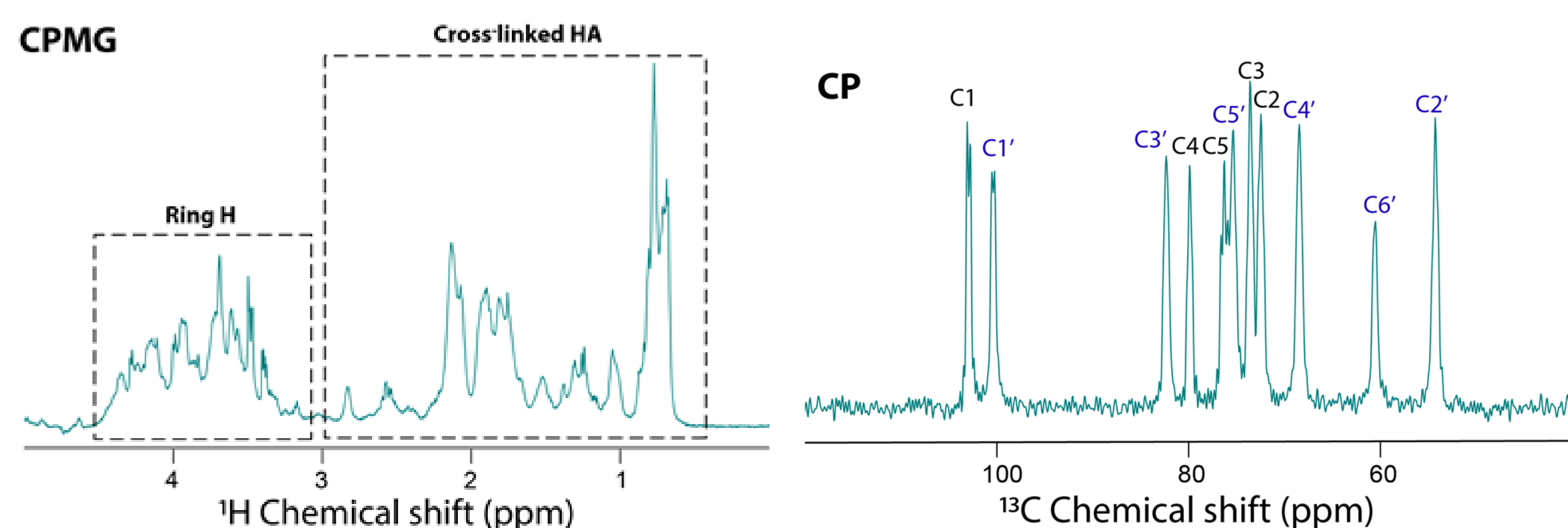
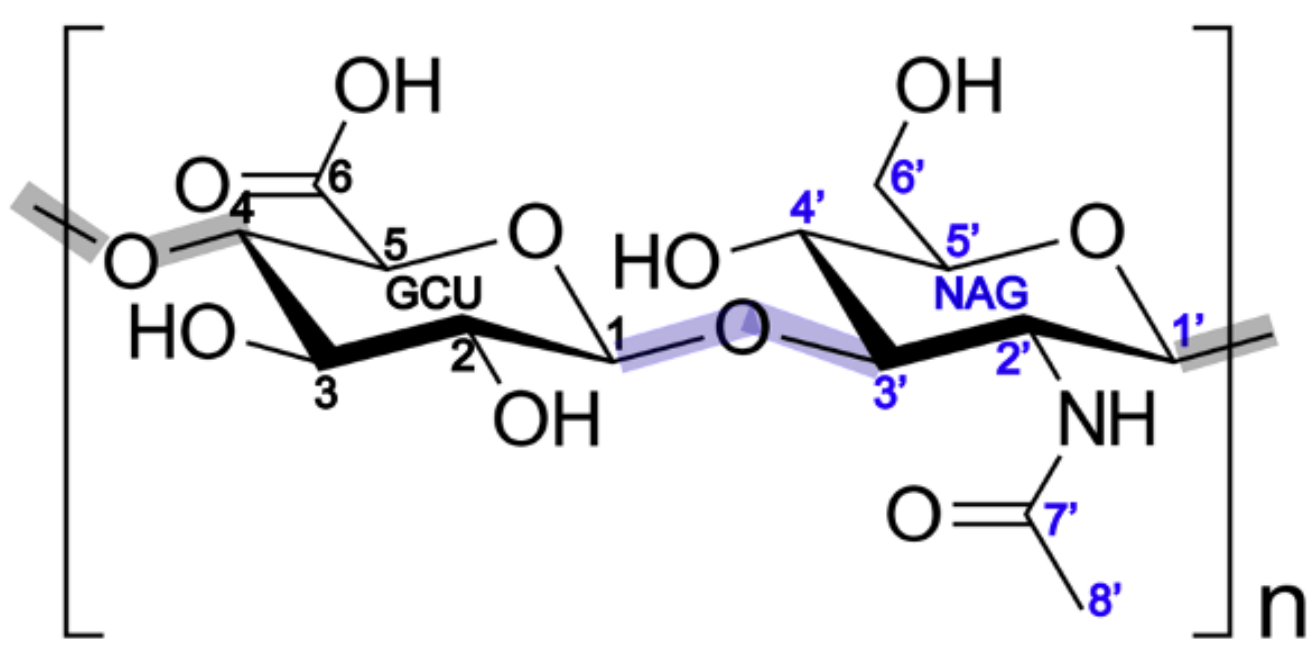
## CMP Spectroscopy with Automation and Broad-Band Channel

Comprehensive multiphase (CMP) NMR probes combine all the electronics and hardware of solution-state NMR (spectrometer lock, susceptibility matched components for sharpest line shape), gel-state NMR (magic angle spinning and magic angle pulsed field gradients) and solid-state NMR (high power circuitry) into a single NMR probe. The CMP probe is now available as H/BB CMP iProbe which adds all the iProbe automation features and a broad-band channel.

With the CMP technology<sup>[1]</sup> all components (liquids, gels, and solids) of samples with mixed phases can be fully differentiated in situ. Furthermore, all NMR studies including molecular interactions (dynamics, bond distances, conformation) and kinetic transport (diffusion, phase change) can potentially be performed.

## Hyaluronic Acid Hydrogel

Hyaluronic acid (HA) is a high molecular weight polysaccharide. HA is a key part of rigid tumor matrices, and is widely used in cosmetics, biomedical nanoparticles and hydrogels. A combination of solution-style and solid-state MAS NMR experiments provided in-depth insights into structure, dynamics and hydration of cross-linked <sup>13</sup>C-HA hydrogels<sup>[2]</sup>. The <sup>1</sup>H and <sup>13</sup>C mono- and bi-dimensional spectra, obtained with remarkable resolution, allowed site-specific assignment of a multitude of immobilized and dynamic species in complex cross-linked hydrogels.

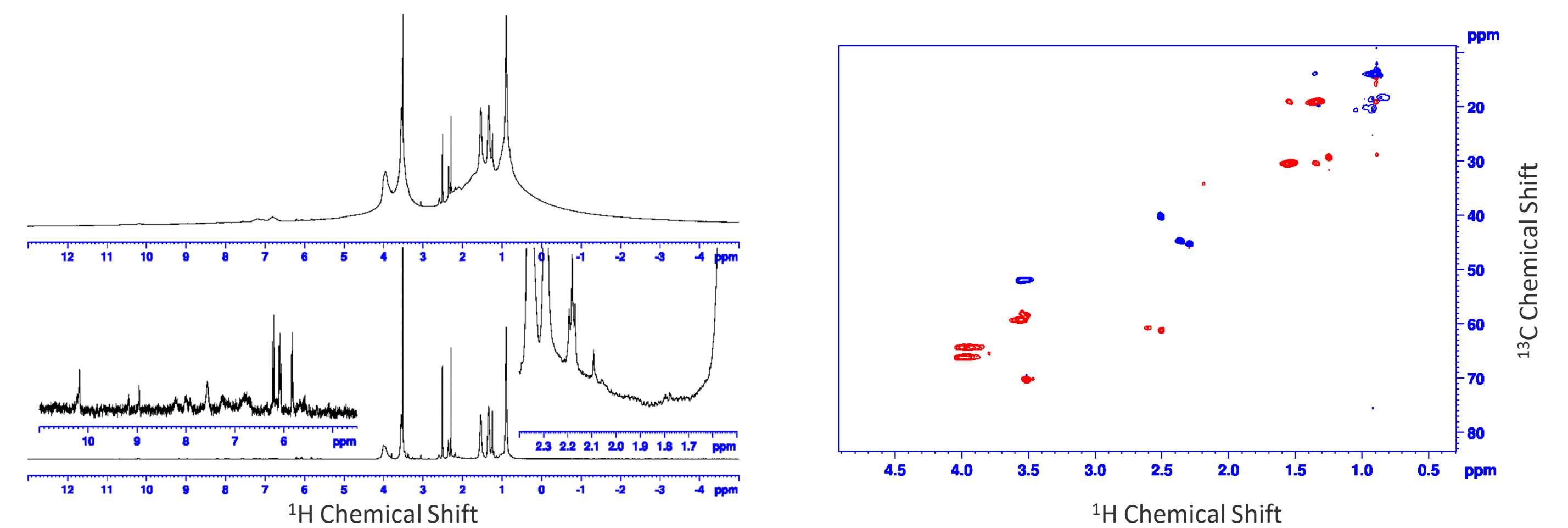


**Fig. 1:** Mono and bi-dimensional <sup>1</sup>H and <sup>13</sup>C spectra of <sup>13</sup>C labeled HA. Top left: the mobile part of the sample is highlighted in this T2-filtered <sup>1</sup>H CPMG experiment. Top right: the <sup>13</sup>C CPMAS experiment select the rigid part of the sample. The bi-dimensional experiment on the bottom left shows trough-space and trough-bond <sup>1</sup>H-<sup>1</sup>H correlations in the mobile phase, while on the left the <sup>13</sup>C-<sup>13</sup>C DARR, based on a CPMAS block, permit to assign all the peaks of the two rings of the repeating units.

## References

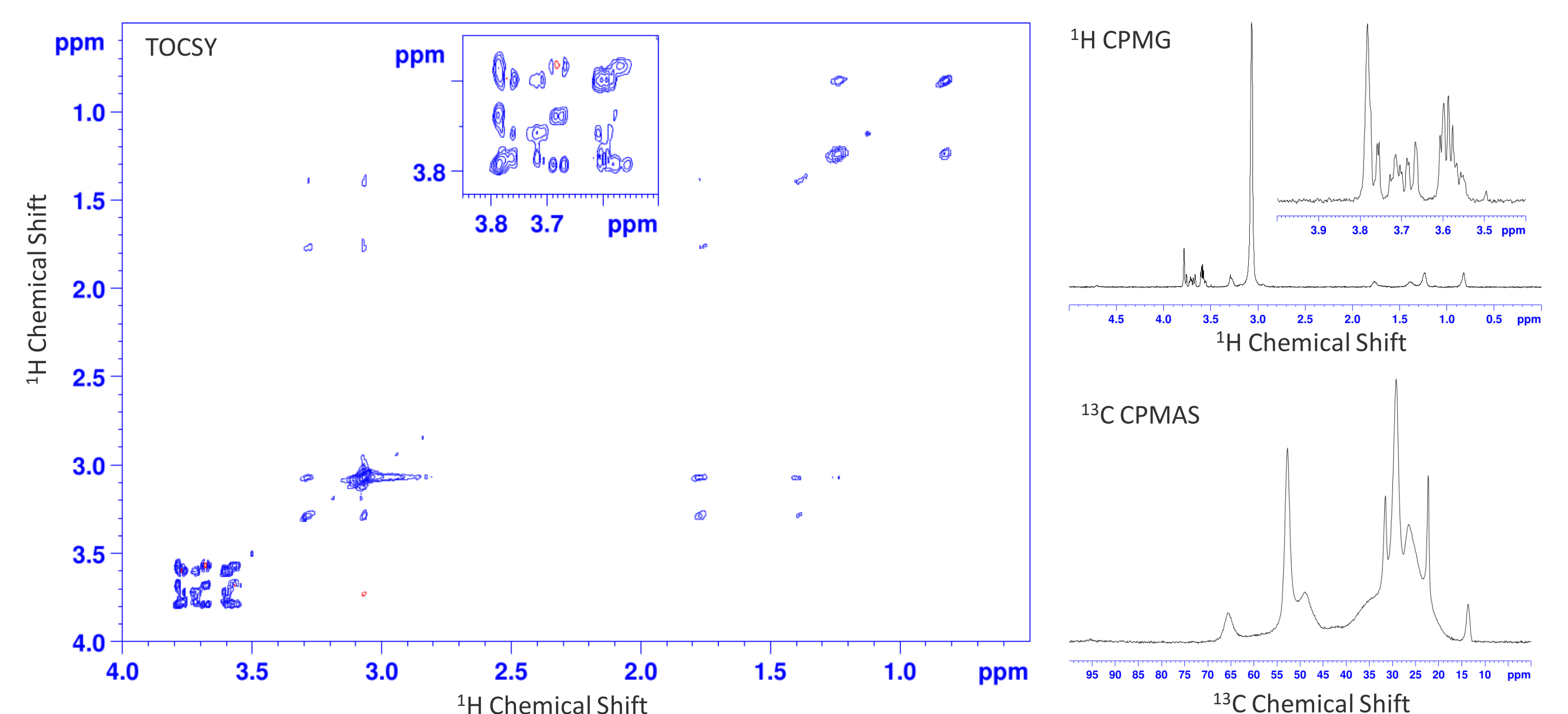
1. D. Courtier-Murias, et al., Comprehensive multiphase NMR spectroscopy: Basic experimental approaches to differentiate phases in heterogeneous samples, *Journal of Magnetic Resonance*, Volume 217, 2012, Pages 61-76, ISSN 1090-7807, <https://doi.org/10.1016/j.jmr.2012.02.009>.
2. Pushpa Rampratap, et al., Production of isotopically enriched high molecular weight hyaluronic acid and characterization by solid-state NMR, *Carbohydrate Polymers*, Volume 316, 2023, 121063, ISSN 0144-8617, <https://doi.org/10.1016/j.carbpol.2023.121063>.

## Latex Acrylic Resin



**Fig. 2:** Spectra of a latex acrylic resin, a low-solubility compound used in paint technologies. Left: <sup>1</sup>H single-pulse and <sup>1</sup>H CPMG spectra, where the more rigid parts of the sample are filtered out, highlighting small peaks in the mobile phase. Right: <sup>1</sup>H-<sup>13</sup>C edited HSQC.

## Polymers as APIs: Colesevelam HCl



**Fig. 3:** Colesevelam-HCl is the leading bile acid binding polymer of the second generation made from crosslinked polyallylamine. Insoluble in all solvents, it can't be studied by traditional solution-state techniques. Left and right top: the <sup>1</sup>H 2D TOCSY and 1D CPMG spectra obtained on the polymer swelled in deuterated water. Bottom right: the <sup>13</sup>C CPMAS spectrum obtained on the dried polymer.

## Summary

The CMP broad-band iProbe is a probe with many functionalities, which allows to perform solution- and solid-state NMR experiments. The two approaches can be combined and mixed, giving valuable insights into heterogeneous samples presenting multiple phases.

## Acknowledgements

We are grateful to Patheon Austria, Linz, Pharma Services / Thermo Fisher Scientific for providing us with the Colesevelam HCl sample.

## CMP Broad-Band iProbe

- Lock, gradients and MAS deliver solution-like spectra even on samples with multiple phases and interfaces
- Robust circuitry and short pulses enables CPMAS-based experiments to select rigid components
- The broad-band channel guarantees a large choice of nuclei
- All automation features of the iProbe family simplify the workflow