MAGNETIC RESONANCE

CMP Broad-band iProbe

Maximum Versatility for MAS Experiments in Mixed Phases

Innovation with Integrity

CMP spectroscopy with iProbe features and broad-band channel

Comprehensive multiphase (CMP) NMR probes combine all the electronics and hardware of solution-state and solid-state NMR into a single NMR probe. Initially designed as single-frequency probes [1], Bruker has since developed the H/BB CMP iProbe, which includes all iProbe automation features as well as a broad-band channel.

Hydrogels

The CMP iProbe enables the in-situ discriminations between the various components (liquids, gels, and solids) within samples exhibiting mixed phases.

Therefore, it is the optimal tool for exploring molecular interactions (including dynamics, bond distances, and conformation) as well as kinetic transport processes (such as diffusion and phase changes).

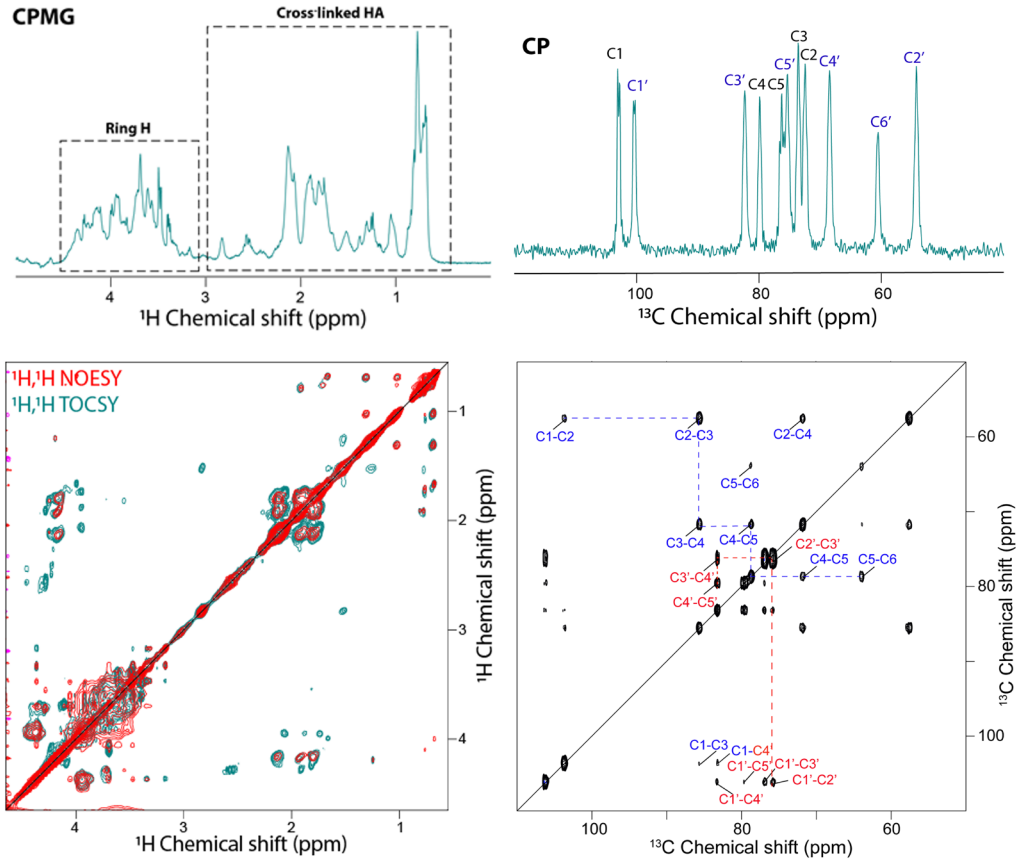

Figure 1: Mono and bi-dimensional ^{1}H and ^{13}C spectra of a gel made from ^{13}C labeled high molecular weight hyaluronic acid (HA). On the top left: the mobile part of the sample is highlighted in this T2-filtered ^{1}H CMPG experiment. On the top right: the ^{13}C CPMAS experiment selects the rigid part of the sample. The bi-dimensional experiment on the bottom left shows trough-space and trough-bond ^{1}H-^{1}H correlations in the mobile phase, while on the left the ^{13}C-^{13}C DARR, based on a CPMAS block, permits to assign all the peaks of the two rings of the repeating units.

Resins and Insoluble Polymers

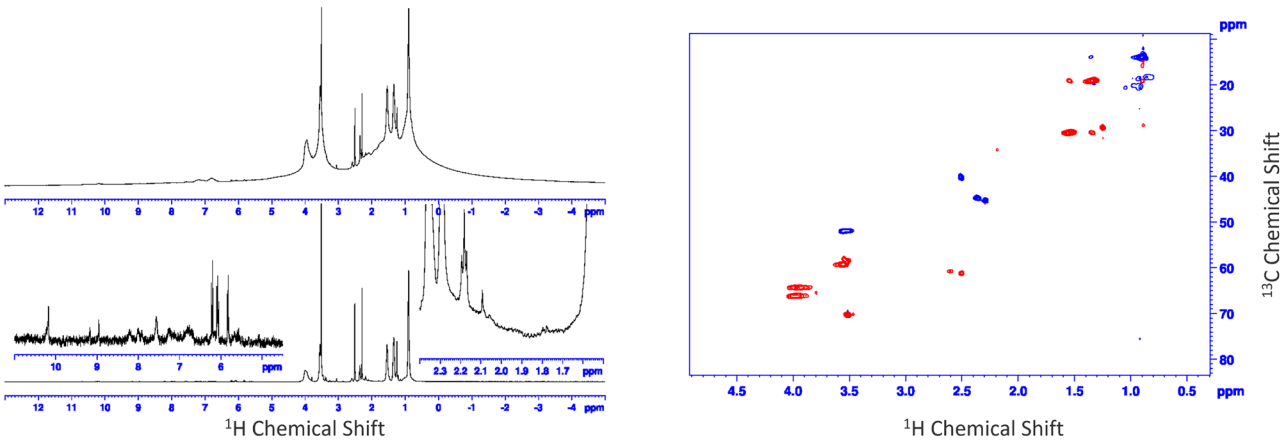

Figure 2: Spectra of a latex acrylic resin, a low-solubility compound used in paint technologies. Left: ^{1}H single-pulse (top) and ^{1}H CMPG (bottom and zooms) spectra, where the more rigid parts of the sample are filtered out, highlighting small peaks in the mobile phase. Right: ^{1}H-^{13}C edited HSQC.

Pharmaceutical Polymers

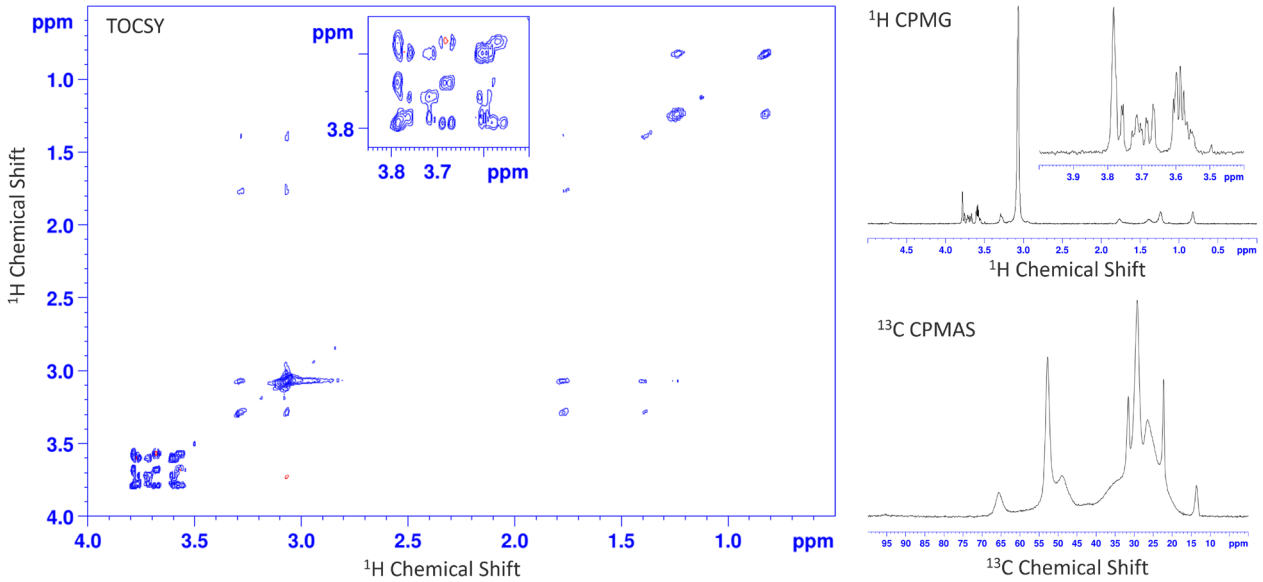

Figure 3: Colesevelam-HCl is the leading bile acid binding polymer of the second generation made from crosslinked polyallylamine. This API is insoluble in all solvents, and therefore cannot be studied by traditional solution-state techniques. On the left and right top the ^{1}H 2D TOCSY and 1D CMPG spectra obtained on the polymer swelled in deuterated water. On bottom right, the ^{13}C CPMAS spectrum obtained on the dry polymer.

Being situated on hydrated biological gels (Figure 1), resins (in Figure 2), or pharmaceutical polymers (Figure 3), the CMP iProbe facilitates a synergistic blend of solution-style and solid-state MAS NMR experiments. The resulting spectra enable precise site-specific assignment of a diverse array of immobilized and dynamic species within intricate matrices. Moreover, it offers profound insights into the structure, dynamics, and hydration of the distinct sample components.

Acknowledgements

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

Summary

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

- 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

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.

Bruker BioSpin
info@bruker.com

bruker.com

Customer Support
https://www.bruker.com/en/services/support.html

Online information
bruker.com/