

¹³C-DOSY-TOSY NMR correlation for In Situ Analysis of Prebiotic Oligosaccharides

E. Vaneckhaute¹, W. De Man², K. Duerinckx¹, J. A. Delcour², F. Taulelle¹, J. A. Martens¹ and E. Breynaert¹

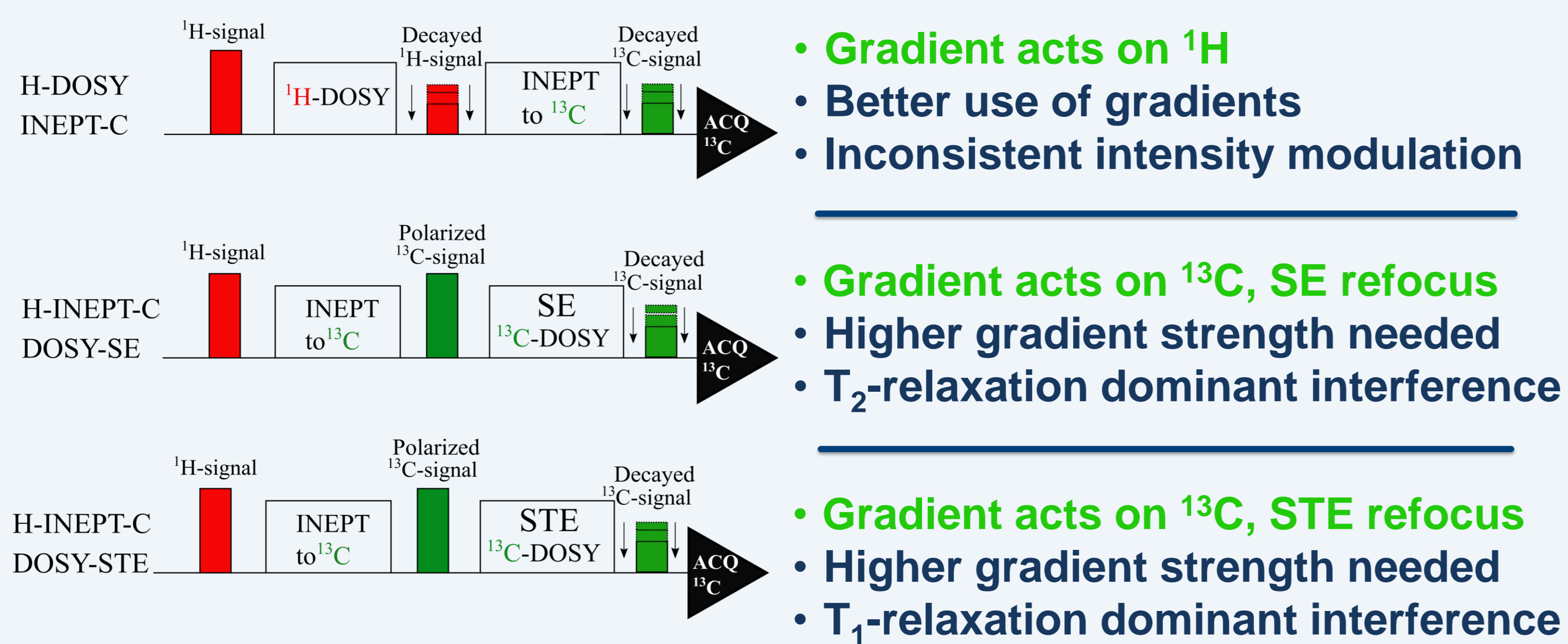
¹ Centre for Surface Chemistry and Catalysis (COK-kat), KU Leuven; ² Laboratory of Food Chemistry and Biochemistry and Leuven Food Science and Nutrition Research Centre (LForCe), KU Leuven



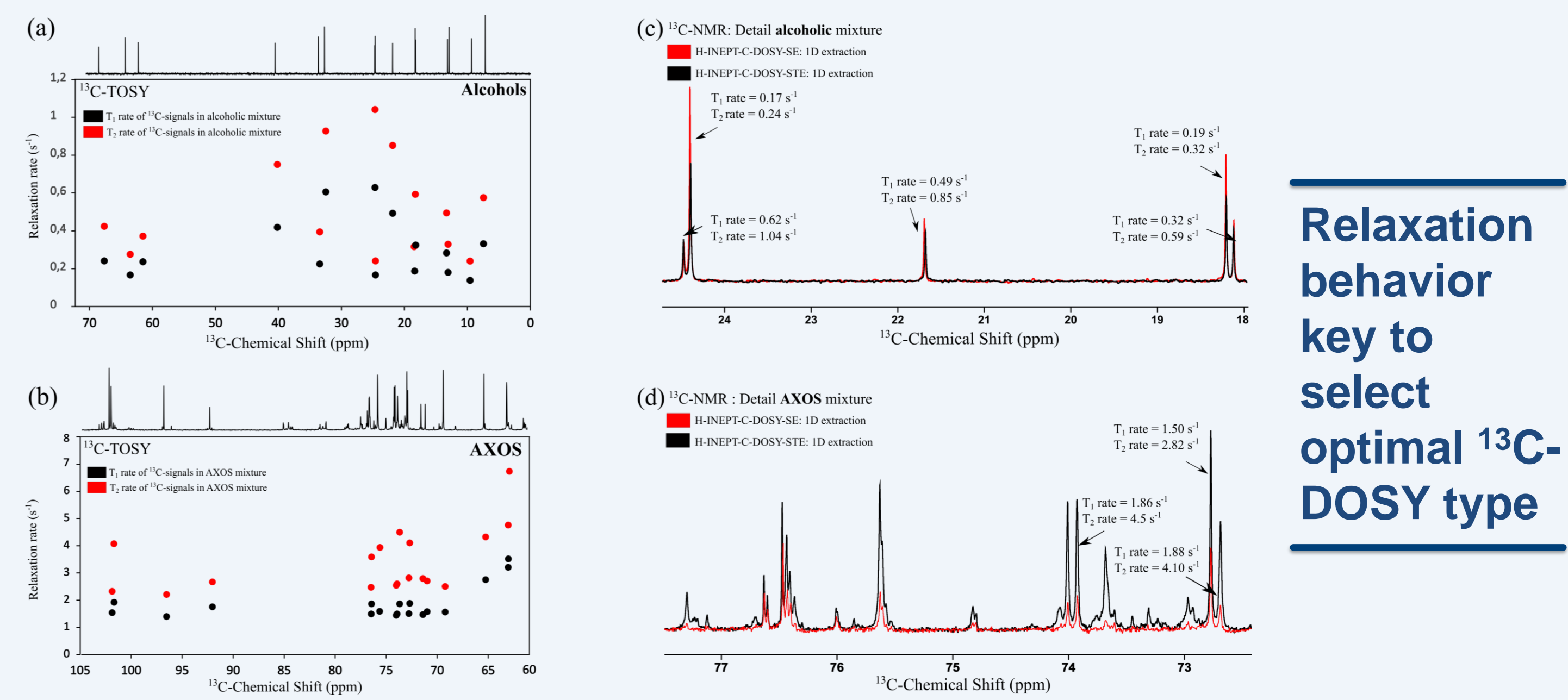
INTRO

Arabinoxylan oligosaccharides (AXOS) are a complex mixture of cereal derived, water soluble prebiotics, obtained by enzymatic hydrolysis of arabinoxylan, a group of dietary fibers exerting numerous nutritional and health-beneficial effects. Such complex biomolecular mixtures are notoriously difficult to characterize without initial physical fractionation. Here we present the *in situ* analysis of AXOS using a variety of state-of-the-art sensitivity-enhanced ¹³C-DOSY methods, enabling virtual separation and identification of the components. Three dimensional correlation plots displaying ¹³C diffusivity (DOSY), relaxation parameters (TOSY) and chemical shift offer a unique way to elucidate the composition of mixtures.

¹³C-DOSY-NMR

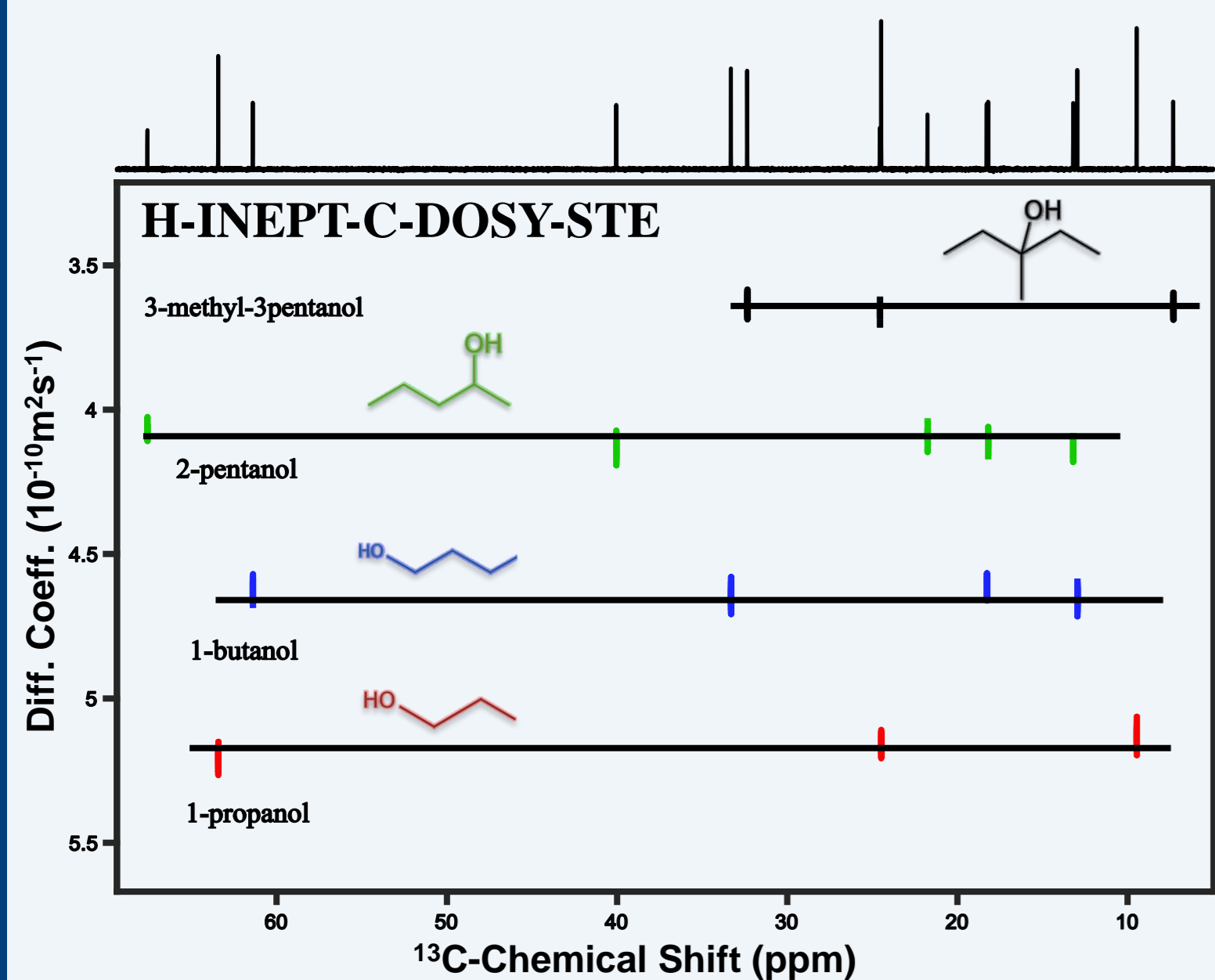


¹³C-TOSY-NMR



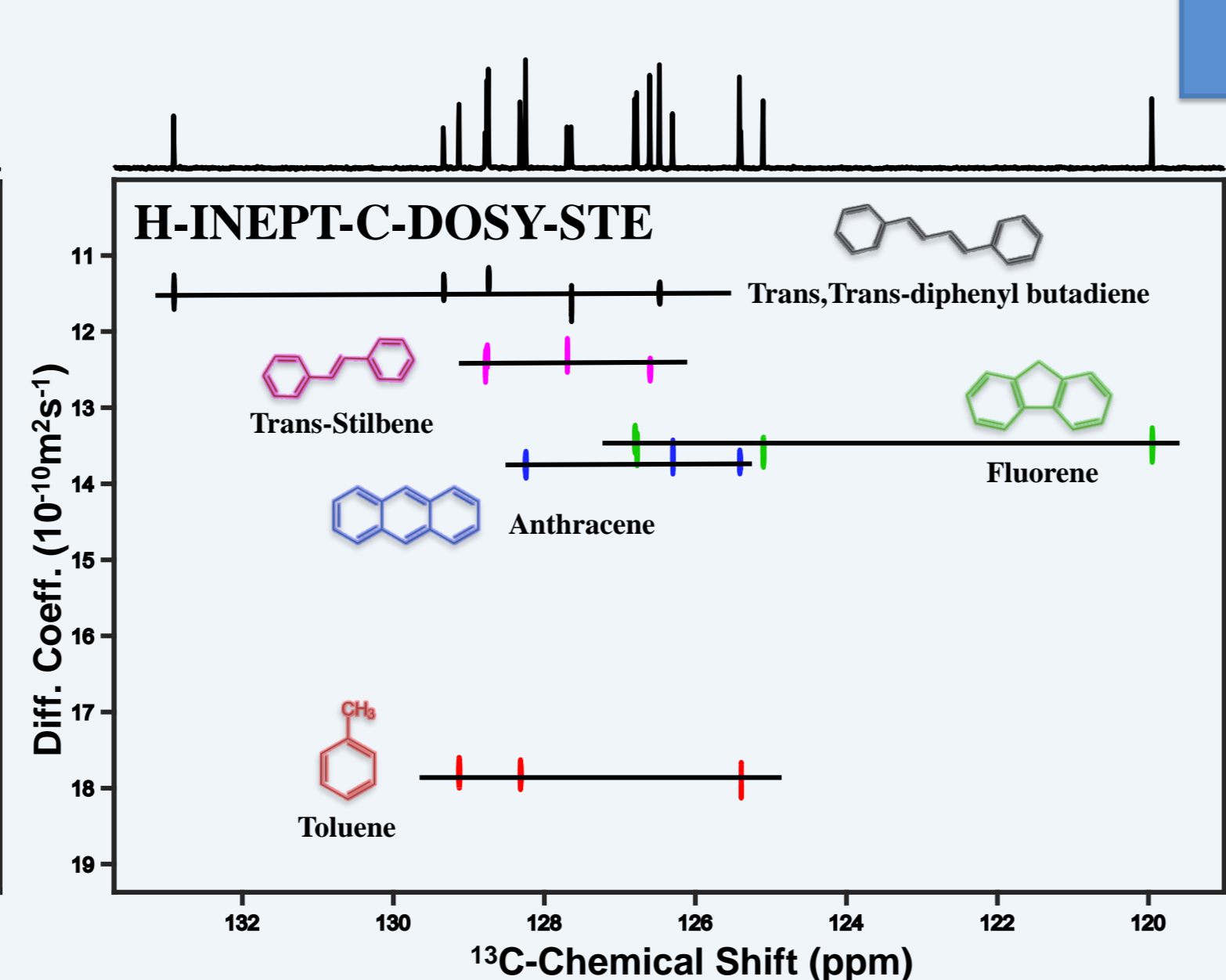
Showcase: Alcohol and Aromatic Mixtures

Alcohol Mixture



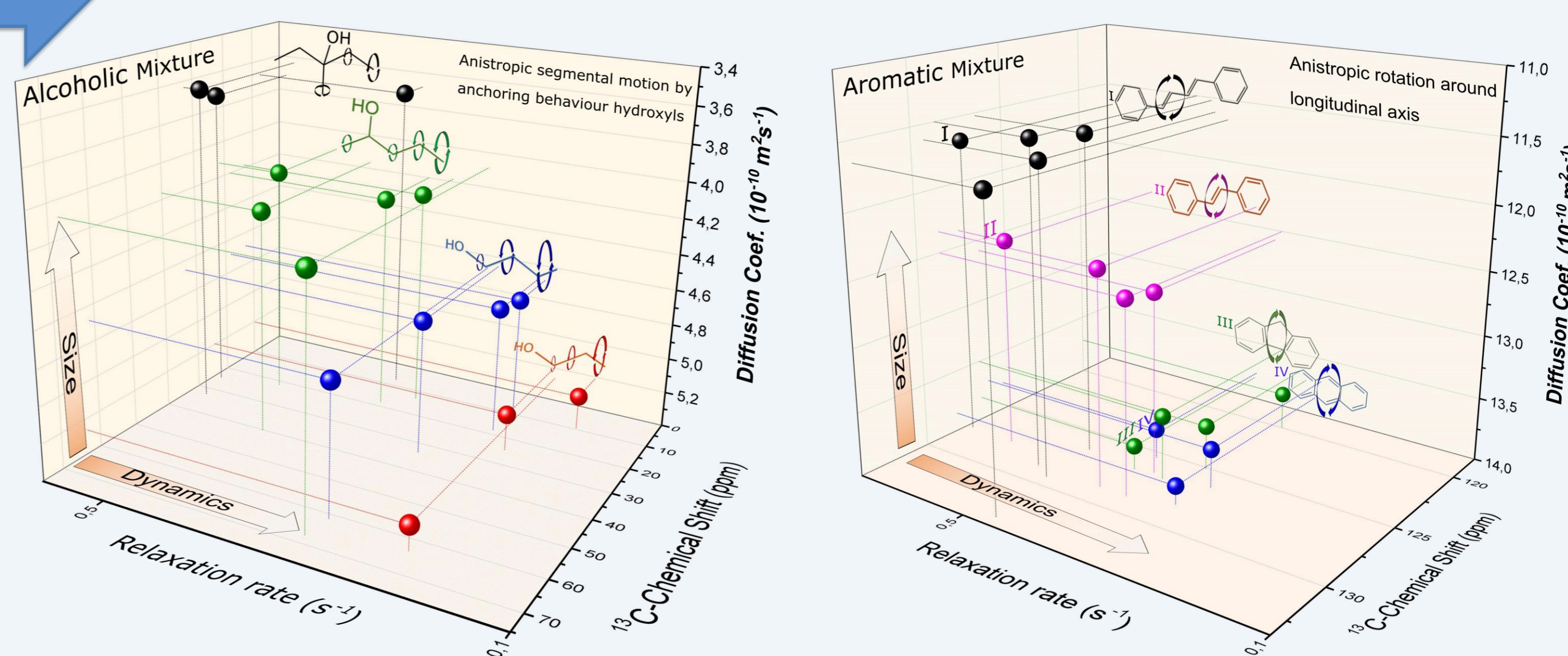
- High resolution in D₂O
- 800 MHz, 10h DOSY

Aromatic Mixture



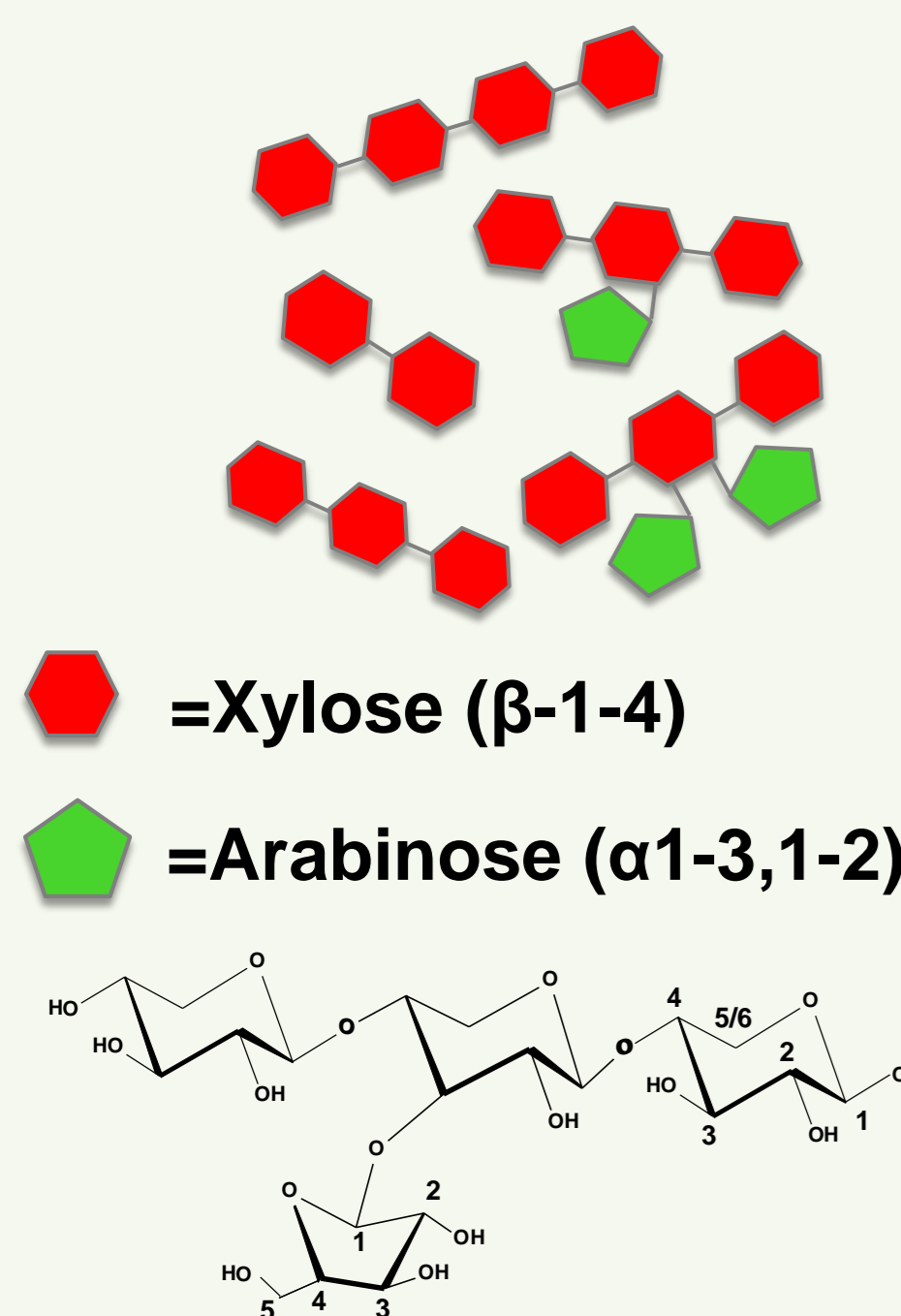
- Limited convection artefacts in CDCl₃
- 800 MHz, 2h DOSY

3D-DOSY-TOSY

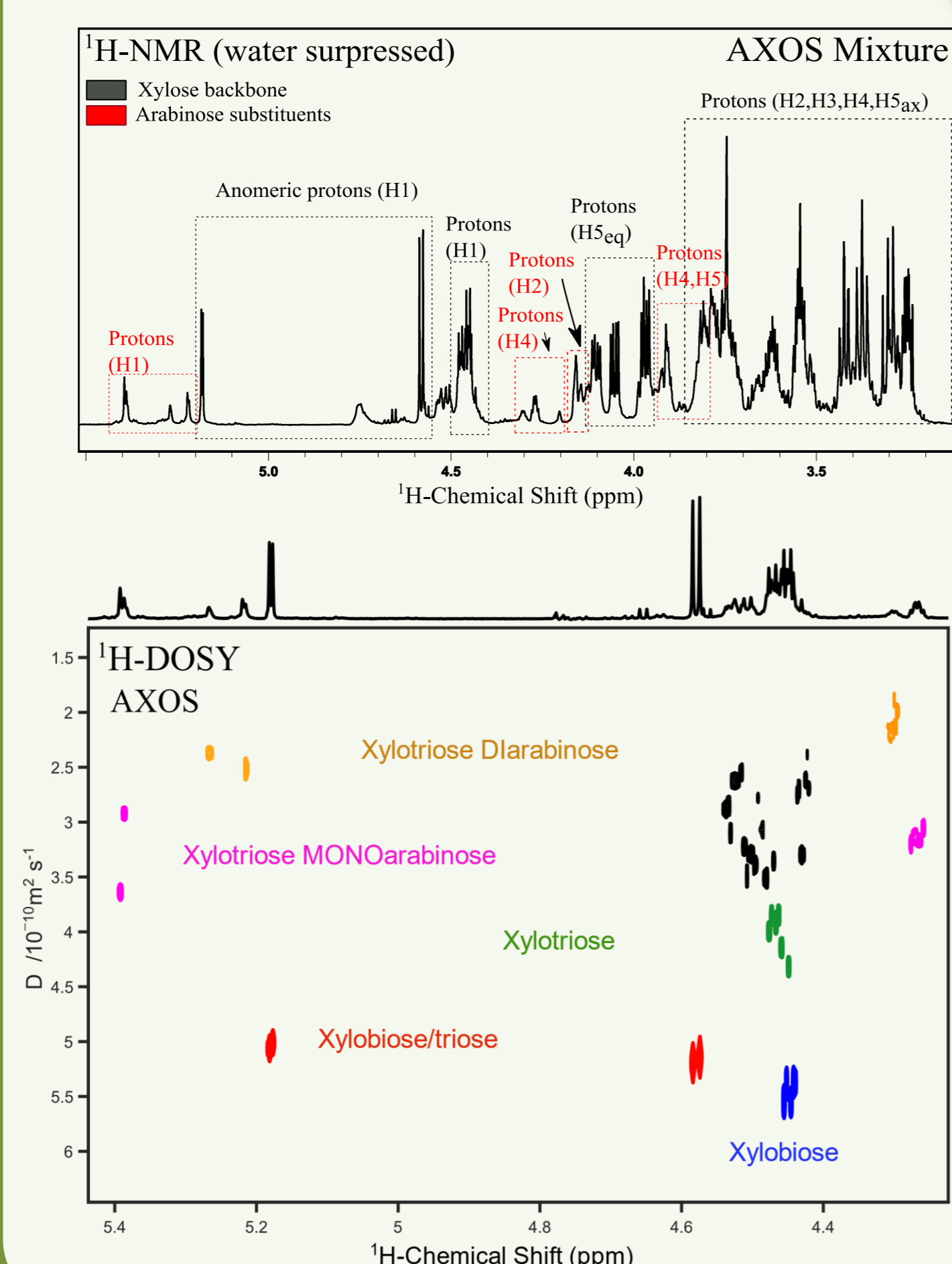


What?

- Prebiotic mixture
- Wheat extract
- Water soluble
- AXOS

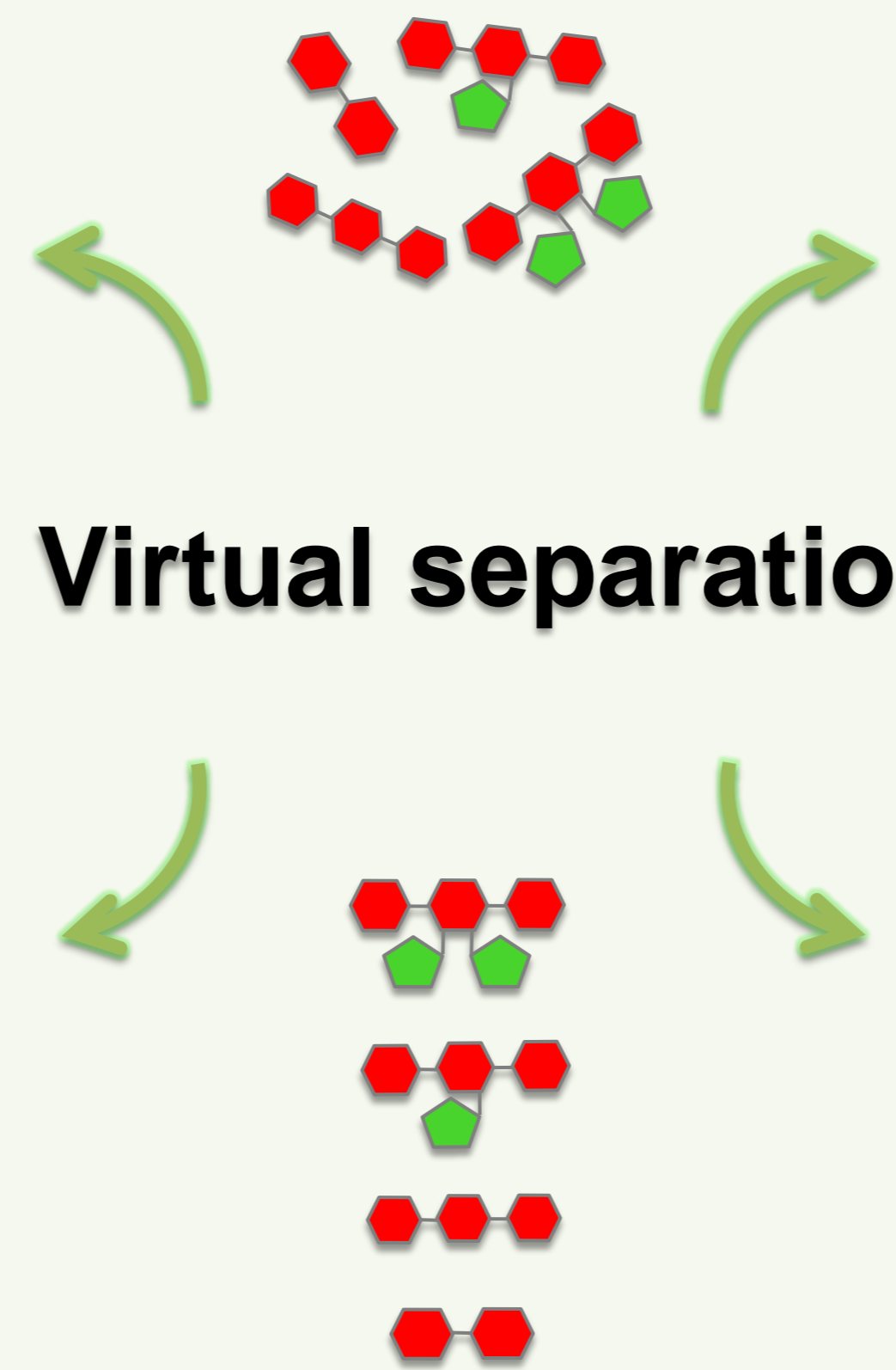


¹H-DOSY

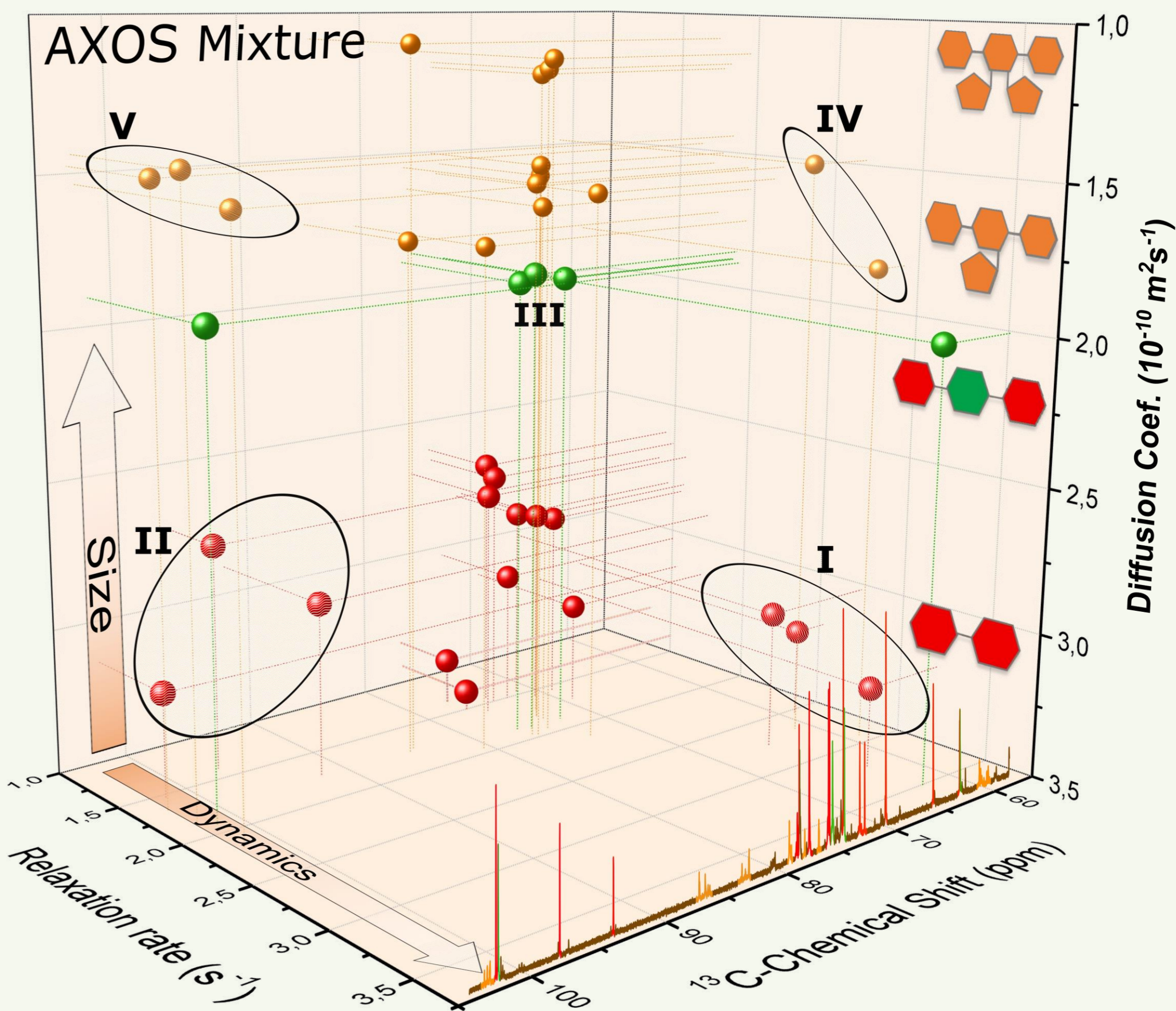


Arabinoxylan Oligosaccharides

Virtual separation



¹³C-DOSY-TOSY



Conclusion

3D-DOSY-TOSY correlation plots combining high resolution ¹³C-DOSY and ¹³C-TOSY allows identification of separate components of an oligosaccharide mixture using size, motion dynamics and chemical functionality as discriminative parameters.