Introducing HR-μMAS NMR probe for μg-scale samples

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NMR has already proven to be a tremendous spectroscopic tool in metabolomics. However, due to its low detection sensitivity, NMR analyses can be challenging especially for mass-limited samples. To overcome this issue, here we introduce a 'new' reliable methodology for profiling µg/nL scale samples based on the Magic Angle Spinning (MAS) technique - High-Resolution µMAS (HR-µMAS). HR-µMAS offers an outstanding opportunity for the detection of metabolites in µg samples with good sensitivity and high spectral resolution, which could widen the existing NMR-based spectroscopy to metabolomic applications and pave the path to its utilization in numerous fields of study.

In comparison to the previous approach with the spinning µ-coil (HR-MACS) [1], HR-µMAS [2] renders improvements in probe practicality, sample-preparation, data repeatability and in the flexibility of NMR experiments. Moreover, we also show the possibility of performing metabolic quantification using HR-µMAS – important analytical component in metabolomics. The excellent NMR spectroscopic capabilities of HR-µMAS shown here for NMR metabolic profiling on µg/nL samples have demonstrated that HR-µMAS could have an impact on future NMR-based metabolomics in biological applications.

[1] Wong, A., Li, X., Molin, L., Solari, F., Elena-Herrmann, B., Sakellariou, D. (2014) µHigh Resolution-Magic-Angle Spinning NMR Spectroscopy for Metabolic Phenotyping of Caenorhabditis elegans. *Anal. Chem.*, 86, 6064–6070.

[2] Duong, N. T., Endo, Y., Nemoto, T., Kato, H., Bouzier-Sore, A.-K., Nishiyama, Y., & Wong, A. (2016). Evaluation of a high-resolution micro-sized magic angle spinning (HRµMAS) probe for NMR-based metabolomic studies of nanoliter samples. *Anal. Methods*, *8*(37), 6815–6820.