

Impact of Magnetic Field Strength on Resolution and Sensitivity of Proton Resonances in Biological Solids

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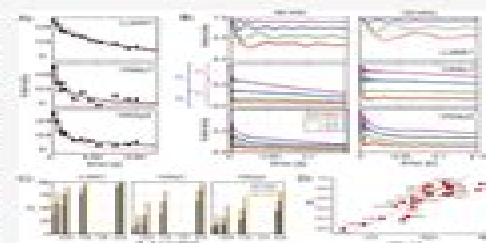


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ABSTRACT: Sensitivity and resolution together determine the quality of NMR spectra in biological solids. Higher magic angle spinning frequencies yield a more efficient suppression of the coupling network and enable atomic-level investigations of protonated protein samples. On the other hand, truncation effects induced by higher magnetic fields have an impact on the achievable sensitivity and resolution. In this work, we address the question of how the proton dipolar coupling network affects the magnetic field strength-dependent gains in sensitivity and resolution. We find that—beyond the canonical $B_0^{3/2}$ dependence—an additional factor of 2 in sensitivity can be achieved for residues embedded in the core of the protein, when the static magnetic field induces a transition from the strong- to the weak-coupling limit. The experiments are carried out using a selectively methyl-protonated ($^{13}\text{CH}_3$) α -spectrin SH3 sample, at magnetic field strengths of 11.75 T (^1H Larmor frequency of 500 MHz) and 23.5 T (^1H Larmor frequency of 1 GHz).



■ INTRODUCTION

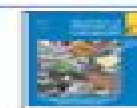
Structure determination of protonated proteins using proton-detected solid-state NMR experiments, acquired at high magnetic fields (1 GHz) and fast (100 kHz) magic angle spinning (MAS), was first demonstrated in 2016.¹ Since then, fast MAS has revolutionized biological solid-state NMR.^{2–5} Fast sample spinning at the magic angle is a prerequisite for proton-detected high-resolution solid-state NMR.⁶ Faster sample spinning averages anisotropic interactions more efficiently, which results in better sensitivity in correlation spectra.⁷ The effect of the MAS rotation frequency on the resolution of amide and methyl proton spectra has been studied recently.^{8,10–12} It has been shown that T_2 of amide protons increases proportionally with the inverse of the rotor period for most residues in a model protein.¹³ As the effective dipole–dipole interaction experienced by methyl protons is much larger than that for any other type of protons in a protein, methyl protons yield the largest line widths, even though the intramethyl dipolar couplings are scaled because of the fast rotation of the methyl group.¹⁴ For a selectively methyl-protonated sample in an otherwise deuterated background, MAS frequencies above 300 kHz are necessary to yield 80% of the maximum attainable signal intensity.¹¹ For MAS frequencies below 70 kHz, $^{13}\text{CHD}_2$ methyl group labeling is necessary to obtain optimal spectral quality. Above an MAS frequency of 70 kHz, $^{13}\text{CH}_3$ isotopomers^{4,15–17} yield the best sensitivity depending on the density of the proton spin system.¹²

The maximum achievable rotation frequency of an MAS rotor is limited by the speed of sound on the rotor surface.¹⁸ Higher rotation frequencies can therefore only be obtained for ever smaller diameter rotors. Lower sample mass is thus traded for faster MAS rates. At first sight, this seems to come at the cost of sensitivity. A 0.7 mm MAS rotor that spins as fast as 110 kHz accommodates effectively less than a milligram of the sample.¹⁹ As the length of an MAS rotor scales approximately linearly with its diameter, the amount of sample in a fast spinning rotor decreases proportionally with r .⁵ On the other hand, the quality factor of the coil and the efficiency of detection increase with smaller coil diameters proportional to $1/r$.²⁰ The apparent coherence decay time T_2^* and thus the signal intensity during proton detection increase with higher MAS frequencies. Longer T_2^* times contribute to the overall intensity linearly with $1/r$.^{9,11,21} Even though the Hartmann–Hahn matching conditions become more selective at high MAS rotation frequencies,^{13,22} $^1\text{H}-T_1$ and T_{ρ} relaxation times increase at faster MAS frequencies which facilitate multi-dimensional solid-state NMR experiments with multiple magnetization transfer steps.⁷ Assuming that polarization transfer contributes another factor proportional to $1/r$ to the

Received: June 15, 2020

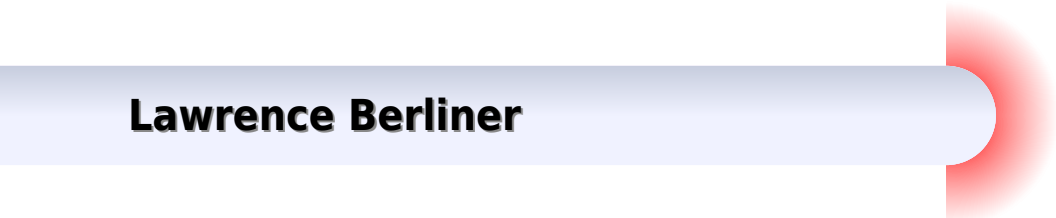
Revised: September 21, 2020

Published: October 1, 2020



Magnetic Resonances In Biological Research

Lawrence Berliner



Magnetic Resonances In Biological Research:

Magnetic Resonances in Biological Research Cafiero Franconi, 1971-01-01 **Magnetic Resonances in Biological Research, Edited by Cafiero Franconi** International Conference on Magnetic Resonances in Biological Research, 3D, Santa Margherita, Cagliari, Italy, 1969, Cafiero Franconi (Ed), 1971 *Biological Magnetic Resonance* Lawrence J. Berliner, Jacques Reuben, 2012-12-06 We are pleased to present Volume 9 of our highly successful series which now celebrates 12 years of providing the magnetic resonance community with topical authoritative chapters on new aspects of biological magnetic resonance As always we try to present a diversity of topic coverage in each volume ranging from applications of in vivo magnetic resonance to more fundamental aspects of electron spin resonance and nuclear magnetic resonance Philip Yeagle presents an eagerly awaited chapter on ^{31}P NMR studies of membranes and membrane protein interactions Alan Marshall has contributed two chapters to the volume one with Jiejun Wu describes magnetic resonance studies of 5S RNA as probes of its structure and conformation the second **Biological Magnetic Resonance** Lawrence Berliner, 2012-12-06 Judging from the articles published in Biochemistry magnetic resonance techniques NMR and ESR are now among the most popular methods in biochemical research The series Biological Magnetic Resonance the fifth volume of which we are proudly presenting is intended to provide authoritative coverage of topics of current interest Previous volumes have covered a number of aspects in a thorough and pedagogical fashion rarely found in other publications in this field Continuing to fulfill the mission of the series this volume presents a chapter by Baxter Mackenzie and Scott on the applications of carbon ^{13}C NMR spectroscopy in investigations of metabolic pathways in vivo Blomberg and Ruterjans give a comprehensive summary of the use of nitrogen ^{15}N NMR in studies of systems of biological interest Phosphorus ^{31}P NMR investigations of enzyme systems are described by Rao Tsai and Bruzik outline the principles of and summarize the state of the art advances in the use of oxygen isotopes ^{17}O and ^{19}F in phosphorus ^{31}P and oxygen ^{17}O NMR studies of biophosphates Lipid protein interactions as reflected in ESR and NMR data are discussed by Devaux We wish to thank the authors for their cooperation in maintaining the and continued high standards of the series **Magnetic Resonance in Biological Systems** A. Ehrenberg, B. G. Malmström, T. Vänngård, 2013-09-24 Magnetic Resonance in Biological Systems Volume 9 is a collection of manuscripts presented at the Second International Conference on Magnetic Resonance in Biological Systems held in Wenner Gren Center Stockholm Sweden on June 1966 The conference is sponsored by International Union of Biochemistry Swedish Medical Research Council Swedish Natural Science Research Council Wenner Gren Center Foundation for Scientific Research This book contains 51 chapters and begins with reviews of NMR investigations of biological macromolecules including proteins amino acids and glycylglycine copper II Considerable chapters are devoted to numerous biological studies using the electronic paramagnetic resonance EPR thus introducing the branch of science called submolecular biology This book also explores other applications of NMR and EPR with special emphasis on blood component analysis and protein metal

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