

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

Kai Xue, Riddhiman Sarkar,^{*} Daniela Lalli, Benita Koch, Guido Pintacuda, Zdenek Tosner, and Bernd Reif^{*}Cite This: *J. Phys. Chem. C* 2020, 124, 22631–22637

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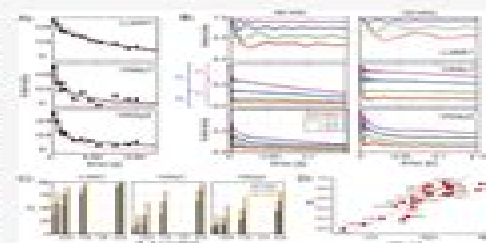


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ABSTRACT: Sensitivity and resolution together determine the quality of NMR spectra in biological solids. Higher magic angle spinning frequencies yield a more efficient suppression of the coupling network and enable atomic-level investigations of protonated 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

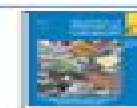
Structure determination of protonated proteins using proton-detected solid-state NMR experiments, acquired at high magnetic fields (1 GHz) and fast (100 kHz) magic angle spinning (MAS), was first demonstrated in 2016.¹ Since then, fast MAS has revolutionized biological solid-state NMR.^{2–5} Fast sample 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.¹⁵ 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 $1/r$.^{4,13,21} 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

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Magnetic Resonances In Biological Research

SJ Ball



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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 *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 complexes. The final chapters survey the principles and applications of Mossbauer spectroscopy. This book will prove useful to analytical chemists and biologists.

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 NMR spectroscopy in investigations of metabolic pathways in vivo. Blomberg and Ruterjans give a comprehensive summary of the use of nitrogen 15 NMR in studies of systems of biological interest. Phosphorus 31 NMR investigations of enzyme systems are described by Rao Tsai and Bruzik. They outline the principles of and summarize the state of the art advances in the use of oxygen isotopes e^{-} 70 and 0 in phosphorus 31 and oxygen 17 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 high standards of the series.

Biological Magnetic Resonance Lawrence J. Berliner, Jacques Reuben, 2012-12-06

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to the volume one with Jiejun Wu describes magnetic resonance studies of 5S RNA as probes of its structure and conformation the second **Energy Research Abstracts**, 1990 *Biological Magnetic Resonance* Lawrence Berliner, 2012-12-06 We have now reached our sixth volume in a series which has somewhat unintentionally become an annual event While we still intend to produce a volume only if a suitable number of excellent chapters in the forefront of biological magnetic resonance are available our philosophy is to present a pedagogical yet critical description and review of selected topics in magnetic resonance of current interest to the community of biomedical scientists This volume fulfills our goals well As always we open the volume with a chapter which directly addresses an in vivo biological problem Phil Bolton's presentation of new techniques in measuring ^{31}P NMR in cells Lenkinski's chapter on the theory and applications of lanthanides in protein studies covers the details highlights and pitfalls of analysis of these complexes in biochemical NMR Reed and Markham summarize the interpretation of EPR spectra of manganese in terms of structure and function of proteins and enzymes Dalton and colleagues describe the applications to biological problems of the relatively new capability of time domain ESR Finally we are pleased to offer a departure from mainstream magnetic resonance with the comprehensive and stimulating chapter by Gus Maki on the theory instrumentation and applications of optically detected magnetic resonance

Spectroscopic Properties of Inorganic and Organometallic Compounds E A V Ebsworth, 2007-10-31 *Spectroscopic Properties of Inorganic and Organometallic Compounds* provides a unique source of information on an important area of chemistry Divided into sections mainly according to the particular spectroscopic technique used coverage in each volume includes NMR with reference to stereochemistry dynamic systems paramagnetic complexes solid state NMR and Groups 13 18 nuclear quadrupole resonance spectroscopy vibrational spectroscopy of main group and transition element compounds and coordinated ligands and electron diffraction Reflecting the growing volume of published work in this field researchers will find this Specialist Periodical Report an invaluable source of information on current methods and applications Specialist Periodical Reports provide systematic and detailed review coverage in major areas of chemical research Compiled by teams of leading experts in their specialist fields this series is designed to help the chemistry community keep current with the latest developments in their field Each volume in the series is published either annually or biennially and is a superb reference point for researchers www.rsc.org/spr *National Library of Medicine Current Catalog* National Library of Medicine (U.S.), 1971 **NMR Nuclear magnetic resonance. in biological research** Kurt Wüthrich, 1976 **Project**

MK-Ultra and Mind Control Technology Axel Balthazar, 2017-05-04 People around the world claim to be victims of mind control technology Medical professionals are quick to marginalize these targeted individuals and diagnose them with mental illness Unfortunately most people are oblivious to the historical precedent for the practice of mind control and the patented technology that exists in the field This book includes a compilation of the government's documentation on MK Ultra the CIA's mind control experimentation on unwitting human subjects all documentation on this program was ordered destroyed by CIA

Director Richard Helms in 1973 but a cache of records survived and were made public through a Freedom of Information Act request in 1977 It also contains over 150 patents pertaining to a wide variety of subjects artificial telepathy voice to skull technology behavior modification through radio frequencies directed energy weapons electronic monitoring implantable nanotechnology brain wave manipulation nervous system manipulation neuroweapons psychological warfare satellite terrorism subliminal messaging and more A must have reference guide for targeted individuals and anyone interested in the subject of mind control technology The experiments and technology described herein reveal the government s activities in a variety of fields utilizing an assortment of methodologies Subjects covered include Drugs Hypnosis Subconscious Isolation Extrasensory Perception Victims Mind Control Technology Artificial Telepathy Behavior Modification Nervous System Manipulation Mind Manipulation Mental Monitoring Directed Energy Weapons Electronic Surveillance Implants and Nanotech Subliminal Messaging and more

Magnetic Resonance Imaging Pottumarthi V. Prasad,2008-02-02 Leading experts in the use of MRI explain its basic principles and demonstrate its power to understand biological processes with numerous cutting edge applications To illustrate its capability to reveal exquisite anatomical detail the authors discuss MRI applications to developmental biology mouse phenotyping and fiber architecture MRI can also provide information about organ and tissue function based on endogenous contrast mechanisms Examples of brain kidney and cardiac function are included as well as applications to neuro and tumor pathophysiology In addition the volume demonstrates the use of exogenous contrast material in functional assessment of the lung noninvasive evaluation of tissue pH the imaging of metabolic activity or gene expression that occur on a molecular level and cellular labeling using superparamagnetic iron oxide contrast agents

Research Awards Index ,1989

Biological Research on Addiction ,2013-05-17 Biological Research on Addiction examines the neurobiological mechanisms of drug use and drug addiction describing how the brain responds to addictive substances as well as how it is affected by drugs of abuse The book s four main sections examine behavioral and molecular biology neuroscience genetics and neuroimaging and neuropharmacology as they relate to the addictive process This volume is especially effective in presenting current knowledge on the key neurobiological and genetic elements in an individual s susceptibility to drug dependence as well as the processes by which some individuals proceed from casual drug use to drug dependence Biological Research on Addiction is one of three volumes comprising the 2 500 page series Comprehensive Addictive Behaviors and Disorders This series provides the most complete collection of current knowledge on addictive behaviors and disorders to date In short it is the definitive reference work on addictions Each article provides glossary full references suggested readings and a list of web resources Edited and authored by the leaders in the field around the globe the broadest most expert coverage available Discusses the genetic basis of addiction Covers basic science research from a variety of animal studies

Research Grants Index National Institutes of Health (U.S.). Division of Research Grants,1975

Fundamentals of Biochemical Pharmacology Z. M. Bacq,2014-05-17 Fundamentals of

Biochemical Pharmacology explains the molecular aspects of drugs and the changes in bio chemical systems The cellular movements that result from such changes are also evaluated Biochemical lesion is extensively defined in the book A discussion on electromagnetic radiation is also provided A chapter of the book is devoted to the principles of electronic and nuclear magnetic resonance The principles and applications of mass spectrometry and combined gas chromatography are then discussed The scientific advances made with the use of immunological methods are the focus of a section of the book Another section provides an introduction to the kinetic properties of reactions made by enzymes The process called homogenization is clearly explained along with a discussion on the use of electron microscopy Autoradiography shows the distribution of compounds at the subcellular level The theoretical background of molecular spectroscopy is presented completely The book is intended for chemists biochemists physicists micro biologists zoologists and botanists

Inorganic and Bio-Inorganic Chemistry - Volume I Ivano Bertini, 2009-02-10 Inorganic and Bio Inorganic Chemistry is the component of Encyclopedia of Chemical Sciences Engineering and Technology Resources in the global Encyclopedia of Life Support Systems EOLSS which is an integrated compendium of twenty one Encyclopedias The Theme on Inorganic and Bio Inorganic Chemistry in the Encyclopedia of Chemical Sciences Engineering and Technology Resources deals with the discipline which studies the chemistry of the elements of the periodic table It covers the following topics From simple to complex compounds Chemistry of metals Inorganic synthesis Radicals reactions with metal complexes in aqueous solutions Magnetic and optical properties Inorganometallic chemistry High temperature materials and solid state chemistry Inorganic biochemistry Inorganic reaction mechanisms Homogeneous and heterogeneous catalysis Cluster and polynuclear compounds Structure and bonding in inorganic chemistry Synthesis and spectroscopy of transition metal complexes Nanosystems Computational inorganic chemistry Energy and inorganic chemistry These two volumes are aimed at the following five major target audiences University and College students Educators Professional practitioners Research personnel and Policy analysts managers and decision makers and NGOs

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