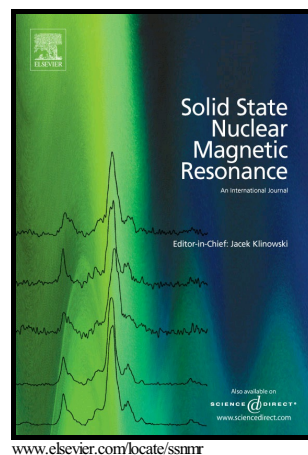


Author's Accepted Manuscript

Broad-band excitation in indirectly detected ^{14}N overtone spectroscopy with composite pulses

Ming Shen, Qun Chen, Jean-Paul Amoureux, Bingwen Hu



PII: S0926-2040(16)30030-3
DOI: <http://dx.doi.org/10.1016/j.ssnmr.2016.05.001>
Reference: YSNMR734

To appear in: *Solid State Nuclear Magnetic Resonance*

Received date: 23 March 2016

Revised date: 6 May 2016

Accepted date: 19 May 2016

Cite this article as: Ming Shen, Qun Chen, Jean-Paul Amoureux and Bingwen Hu, Broad-band excitation in indirectly detected ^{14}N overtone spectroscopy with composite pulses, *Solid State Nuclear Magnetic Resonance* <http://dx.doi.org/10.1016/j.ssnmr.2016.05.001>

This is a PDF file of an unedited manuscript that has been accepted for publication. As a service to our customers we are providing this early version of the manuscript. The manuscript will undergo copyediting, typesetting, and review of the resulting galley proof before it is published in its final citable form. Please note that during the production process errors may be discovered which could affect the content, and all legal disclaimers that apply to the journal pertain.

Broad-band excitation in indirectly detected ^{14}N overtone spectroscopy with composite pulses

Ming Shen, Qun Chen, Jean-Paul Amoureux, Bingwen Hu

Shanghai Key Laboratory of Magnetic Resonance, School of Physics and Materials Science, East China Normal University, Shanghai, 200062

shen.ming@outlook.com

bwhu@phy.ecnu.edu.cn

Abstract

We show here that composite pulses allow broad-band excitation of nitrogen-14 overtone frequencies through proton detected *D*-HMQC experiment (referred to $^1\text{H}\{-^{14}\text{N}_{\text{OT}}^{\text{DQ}}\}$ *D*-HMQC). Experimental verifications have been performed on glycine, L-histidine and N-acetyl-valine (NAV) samples. Composite pulses enable symmetric excitations of ^{14}N sites with large shift differences. Therefore, this approach is promising for recording high resolution $^1\text{H}\{-^{14}\text{N}_{\text{OT}}^{\text{DQ}}\}$ *D*-HMQC spectra of most amino-acids, pharmaceutical samples and peptides.

Key words

Solid-state NMR, ^{14}N overtone spectroscopy, proton-detected *D*-HMQC

Introduction

Solid-State NMR (SS-NMR) is a powerful tool for probing structural and dynamic information in biomolecules. ^{13}C and ^{15}N enrichments are often utilized to provide local binding and distance constraints with atomic resolution. SS-NMR study of ^{14}N nucleus is less common due to its spin-1 value and its large quadrupole interaction that broadens the signal. However, ^{14}N SS-NMR can provide unique information on electric field gradients, which can provide detailed information on structure and dynamics at the molecular level. Although experimentally demanding, the direct 1D detection of nitrogen-14 single-quantum transitions, $^{14}\text{N}^{\text{SQ}}$, is nevertheless feasible under both static [1,2] and magic angle spinning (MAS) [3] conditions. However, due to the overlap of the broad line-shapes from different sites, $^{14}\text{N}^{\text{SQ}}$ directly-detected 1D spectra with many ^{14}N sites are quite difficult to analyse. As a result, the dipolar-assisted heteronuclear multiple quantum coherence (*D*-HMQC) MAS method is often used to obtain high-resolution signals of ^{14}N nuclei.[4,5] In *D*-

Download English Version:

<https://daneshyari.com/en/article/5420248>

Download Persian Version:

<https://daneshyari.com/article/5420248>

[Daneshyari.com](https://daneshyari.com)