

Quantitative Evaluation of ^{13}C NMR Peak Intensity for Polyolefin Solution.

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The ^{13}C -NMR is an essential tool to obtain primary structure of polyolefins. It provides not only monomer composition but monomer sequence distribution and tacticity. To obtain quantitative ^{13}C NMR spectra, the inverse gated decoupling (IGD) method is frequently employed to avoid steady state NOE since the degree of enhancement based on NOE varies according to different carbons in the ^{13}C NMR signal intensity. Due to the lower sensitivity and longer relaxation time, ^{13}C NMR experiment takes very long time to obtain a spectrum with adequate signal-to-noise ratio. With IGD method, much longer time is needed to recover sensitivity loss by abolishment of unwanted NOE in exchange for quantitative results. Relaxation agents are employed to make recycle time shorter and make signal acquisition more time-efficient¹⁾.

The sensitivity can be enhanced with DEPT and INEPT approaches by transferring polarization from $^1\text{H}(\text{I})$ to $^{13}\text{C}(\text{S})$, but the enhancements for different spin systems are not uniform and quantitative analyses are seriously affected, since the enhancements depend on coupling constants ($^1J_{\text{SI}}$) and spin system SI , SI_2 , SI_3 . Jing et al.²⁾ reported a quantitative POMMIE method optimized for organic compounds with a large J-coupling range (90-230 Hz), and the ^{13}C signals for all spin systems are nearly equally enhanced with it. Since polyolefins exhibit almost unique coupling constants ($^1J_{\text{SI}} = \text{ca.} 125 \text{ Hz}$) regardless spin system, the uniform enhancements can be achieved more effectively by choosing experimental parameters that are suitable for polyolefins. In this report a quantitative POMMIE optimized for polyolefins is applied for quantitative analysis of α -olefin homo- and co-polymers as an alternative method to IGD method.

References:

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2. B. Jing, N. Xiao, Z. Zhou, X. Mao, M. Liu, *Anal. Chem.*, **2008**, 80, 8293-8298.