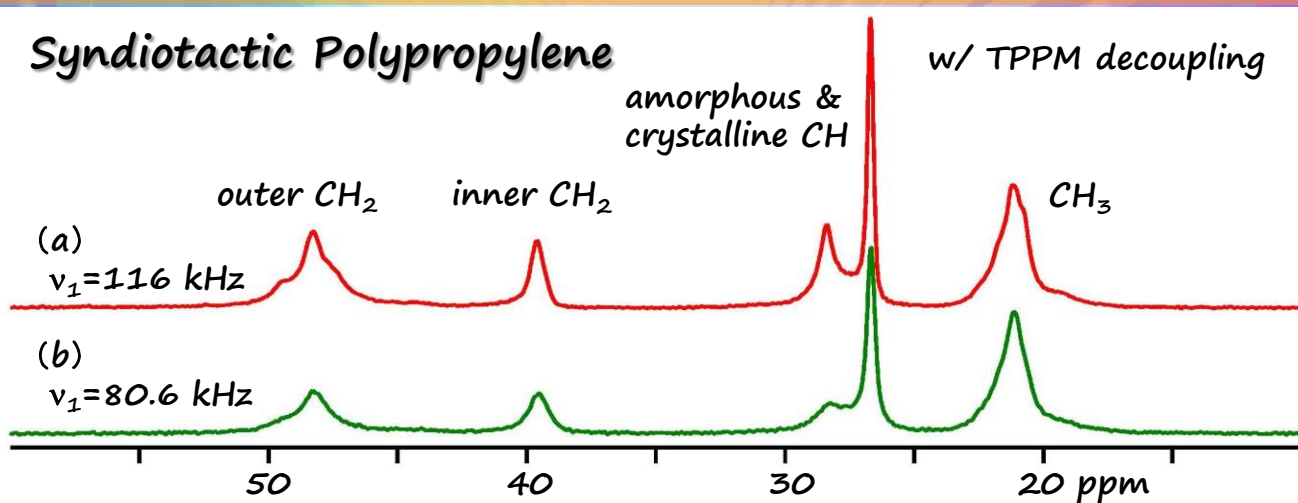


# The Effects of $^1\text{H}$ Decoupling Strength on $^{13}\text{C}$ MAS Spectral Resolution for Solid Polymer Samples

High resolution  $^1\text{H}$  decoupled  $^{13}\text{C}$  MAS spectra (CPMAS and DDMAS spectra), exhibiting shoulders and splittings, may yield the information on tacticity distribution and sample heterogeneity. Figure shown below is  $^{13}\text{C}$  CPMAS spectra for syndiotactic polypropylene, observed with (a)  $^1\text{H}$  decoupling RF field strength  $\nu_1$  of 116kHz and (b)  $\nu_1$  of 80.6kHz.



Obviously, strong  $^1\text{H}$  decoupling irradiation improves spectral resolution: In (a), one can recognize distinct shoulders for outer  $\text{CH}_2$  and  $\text{CH}_3$ , as well as a conspicuous splitting for CH.

$^{13}\text{C}$ - $^1\text{H}$  dipolar interaction  $\nu_{\text{CH}}$ , ca 20kHz, can be averaged out by  $^1\text{H}$ - $^{13}\text{C}$  heteronuclear decoupling  $\nu_1$ , whether 80kHz or 120kHz. However, for most organic solid samples, "the third nucleus" of  $^1\text{H}$  in addition to  $^{13}\text{C}$  and  $^1\text{H}$  lowers  $^{13}\text{C}$  spectral resolution.

$\text{CH}_2$  as a three-spin model system involves not only two  $^{13}\text{C}$ - $^1\text{H}$  dipolar interactions of  $\nu_{\text{CH}} = 20\text{kHz}$  but also  $^1\text{H}$ - $^1\text{H}$  dipolar interaction of  $\nu_{\text{HH}} = 20\text{kHz}$ .  $^1\text{H}$ - $^1\text{H}$  dipolar interaction cannot perfectly be eliminated using multiple pulse techniques and lowers  $^{13}\text{C}$  spectral resolution through high-order perturbation cross terms with  $^1\text{H}$  chemical shift anisotropy. On the contrary,  $^1\text{H}$ - $^1\text{H}$  dipolar interaction left without manipulation would be removed via "self-decoupling" phenomena, but MAS prevents such self-decoupling. TPPM decoupling may recouple MAS-reduced  $^1\text{H}$ - $^1\text{H}$  dipolar interaction, thereby improving  $^{13}\text{C}$  spectral resolution via self-decoupling. Reference: P. Hodgkinson, Prog. NMR Spect., **46**, 197-222 (2005).

