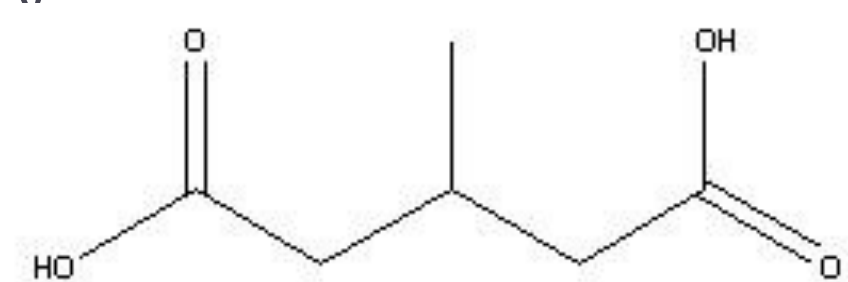


Introduction

- High Resolution-creating experimental conditions that produce narrow spectral lines
- Enhances signal
 - Increases signal-to-noise ratio since peak heights are increased when line widths are reduced
- To acquire data the spectrometer must first be optimized
 - Methylglutaric Acid (MGA)



High Powered Decoupling

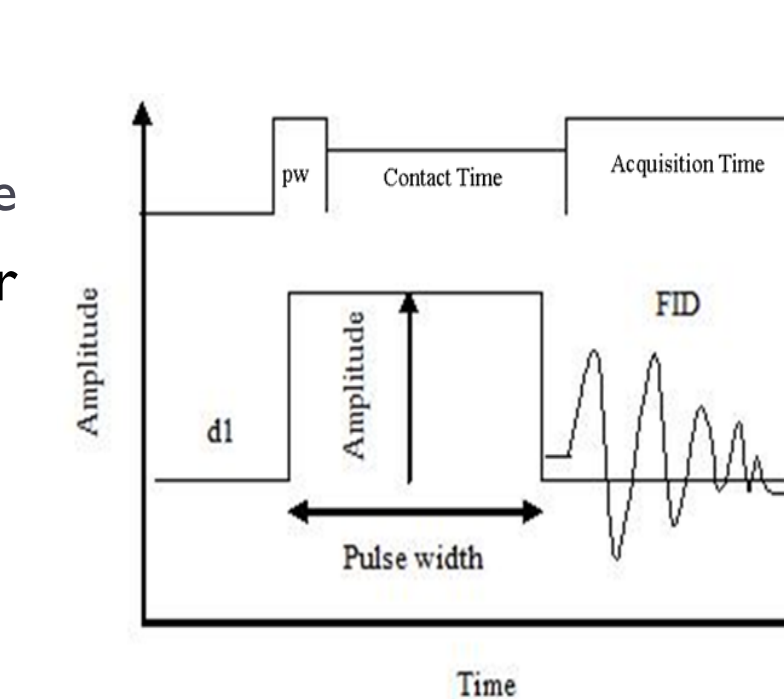
- Direct dipolar coupling
 - Broadening due to local magnetic field of neighboring nuclei
 - Averages out in liquids due to motion
- Proton-Carbon dipolar coupling is strong
 - Directly bonded protons can produce couplings as large as 40KHz
 - To remove, MAS rate must exceed dipolar coupling
- Irradiate the sample at the proton's resonance frequency while acquiring ^{13}C signal
 - NMR coil needs ~300 watts to achieve 100KHz RF

SPE Variables

- dI-delay that allows nuclei to reach equilibrium in magnetic field
- Amplitude - strength of pulse
- Pulse width-duration of excitation
 - 90° pulse width
 - pulse that tips magnetization from the z axis into the xy plane
- Free induction decay (FID)-oscillating voltage induced in coil from the sample. NMR signal that is digitized by the receiver.

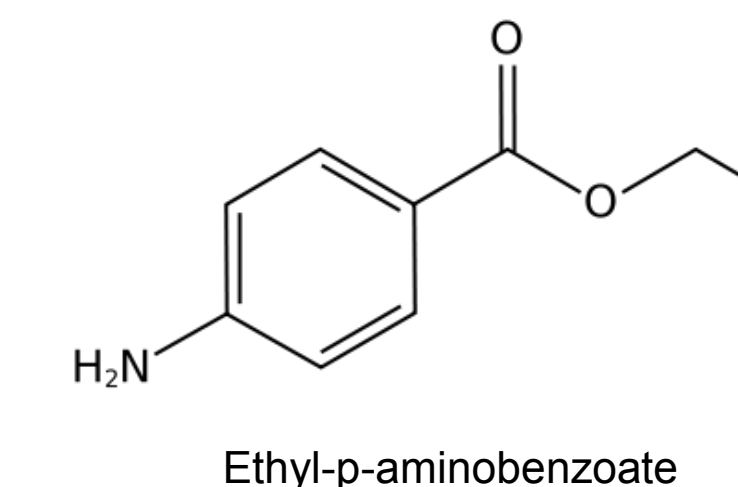
Cross Polarization

- Allows for transfer of magnetization
 - Proton to Carbon
 - Occurs during contact time
- Increases signal by a factor of 2
- Allows for increased rate of pulsation
 - T_1 of ^{13}C is often at least 10 times that of ^1H



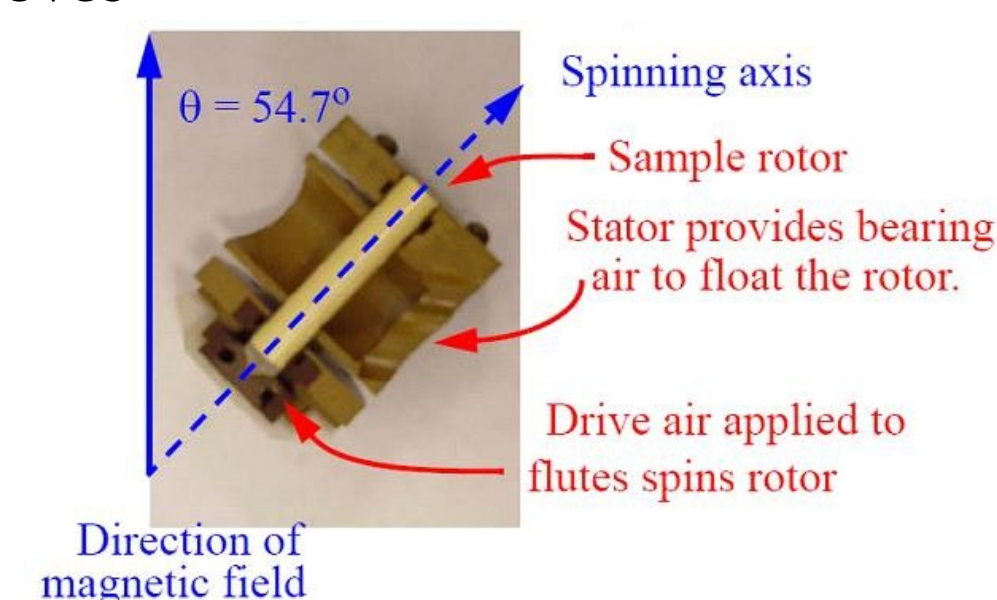
Acquire Data

- Spectrometer is now ready to acquire high resolution solid-state NMR spectra of organic solids

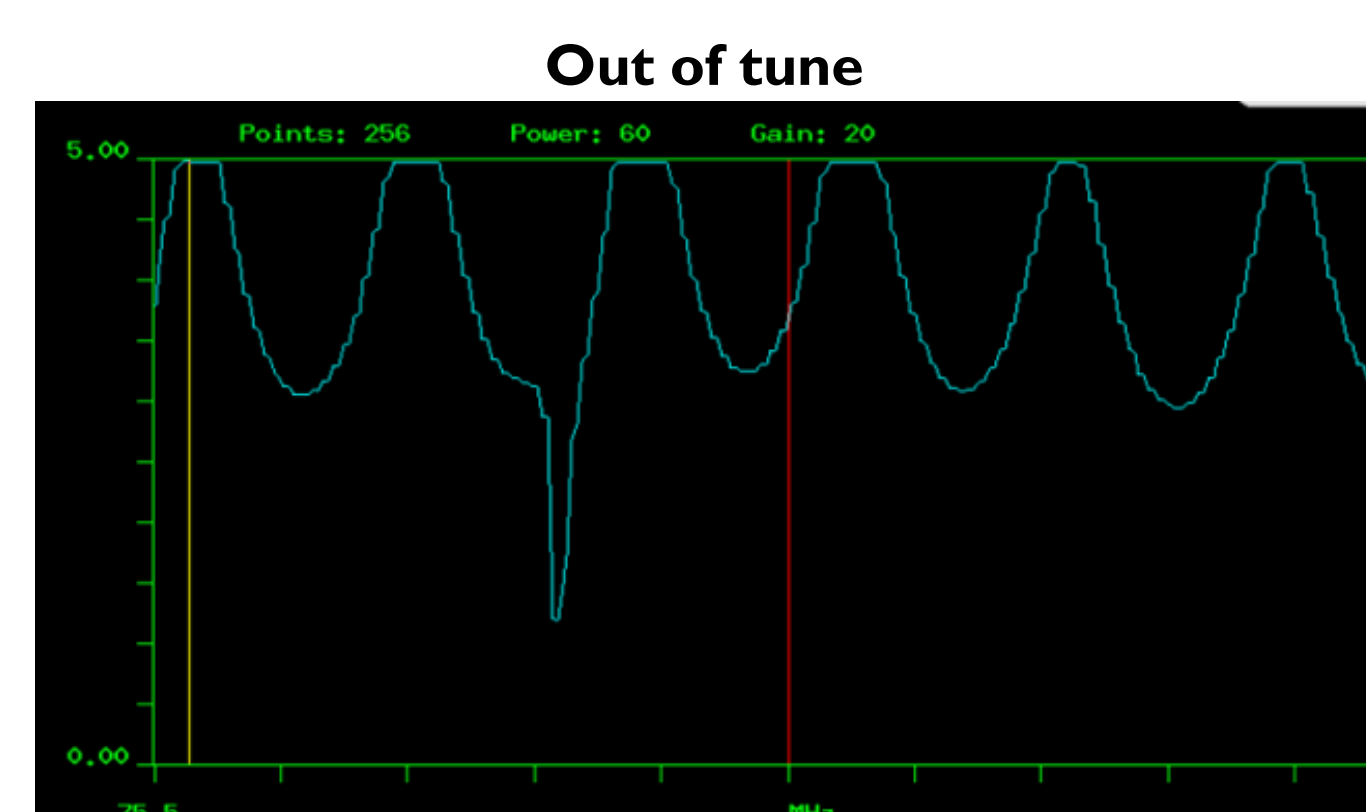


Magic Angle Spinning

- Magic Angle: 54.73°
 - Related to Legendre's second associated polynomial
- Provides mechanical averaging of sample with respect to external field
- Dramatically improves spectrum

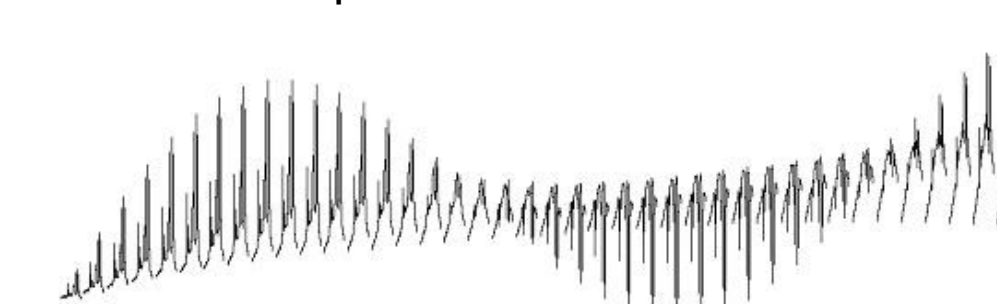


Graphical Tuning



Pulse Width

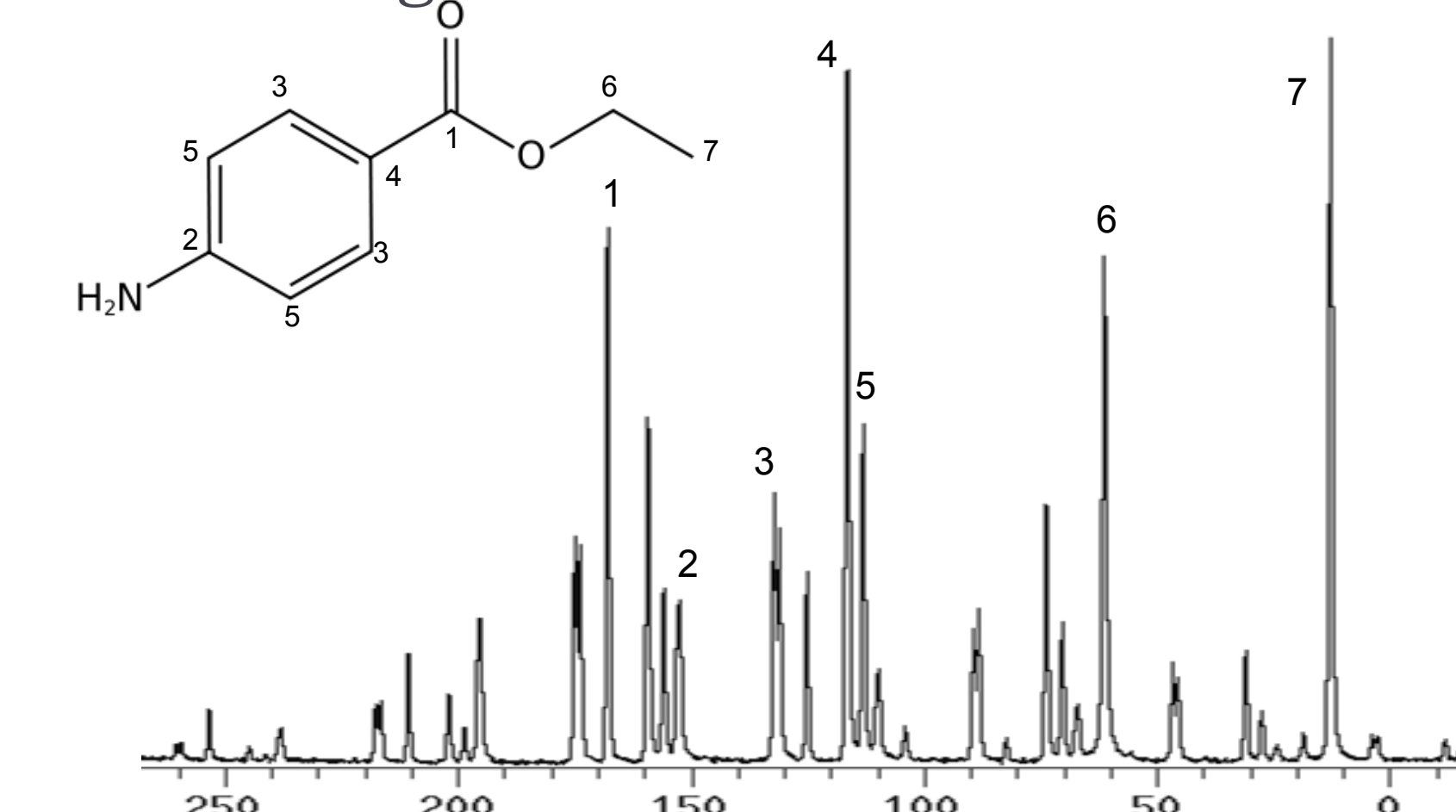
- ^1H is easiest to observe
 - Broad signal may cause baseline distortion
- Peak intensity should form sine wave for both the ^1H and ^{13}C arrays
 - Easier to observe 180° than 90°
 - 180° occurs at first null
 - Half of this value ~ 90° pulse width



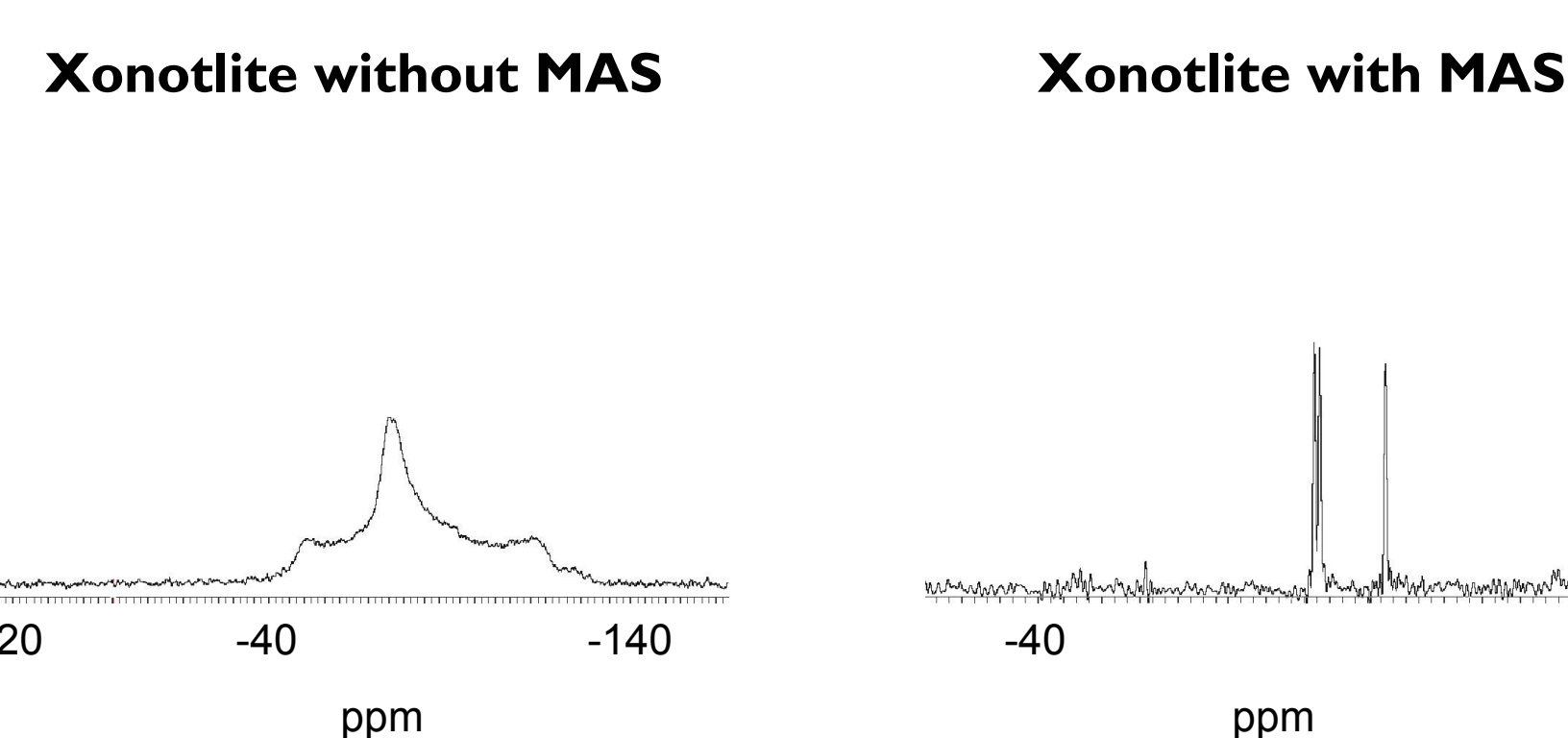
Sample Dependent Parameters

- Pulse delay (dI)-determined by calculating T_1
- T_1 is the decay constant for the z component of the nuclear spin magnetization
- Optimal pulse delay is 1.26 times the T_1 value
- Contact time-period in which magnetization is transferred from ^1H to ^{13}C

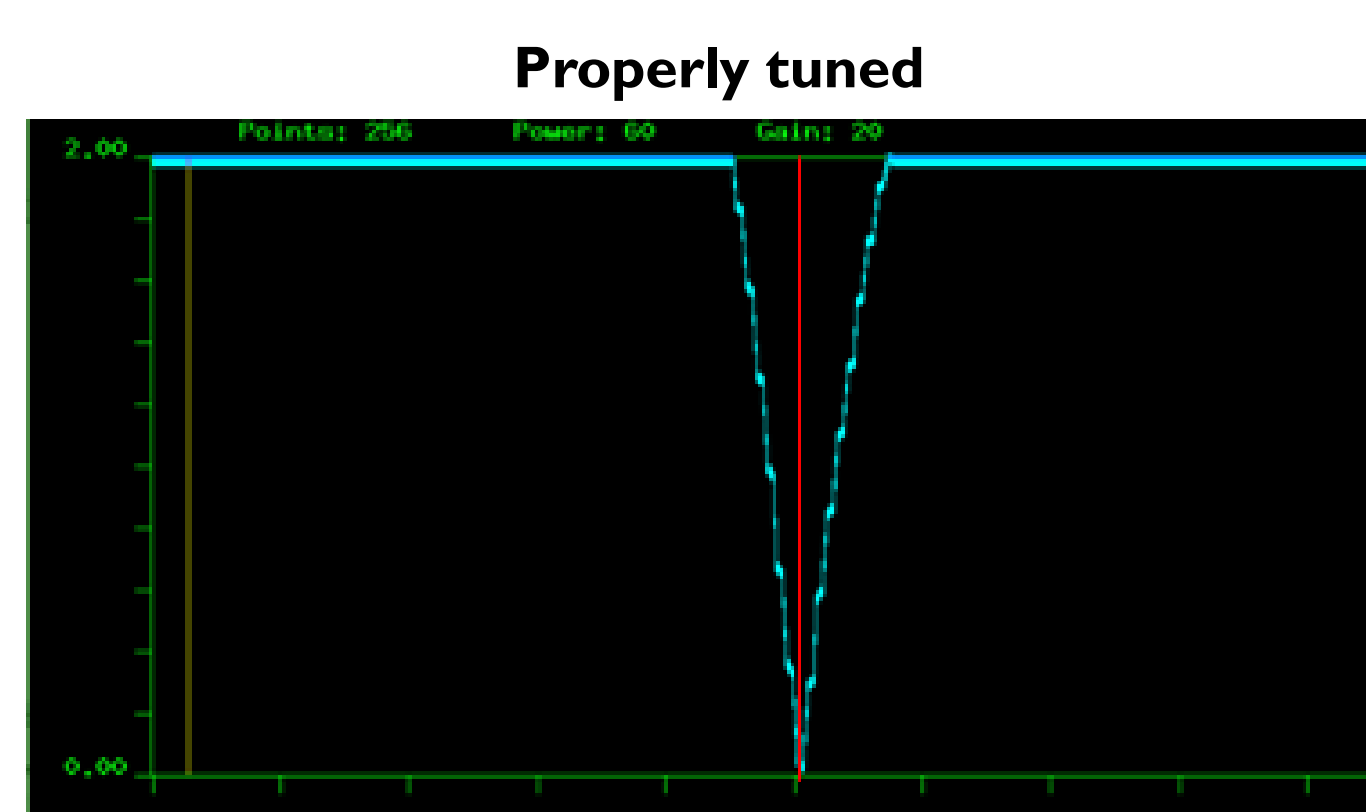
Peak Assignment



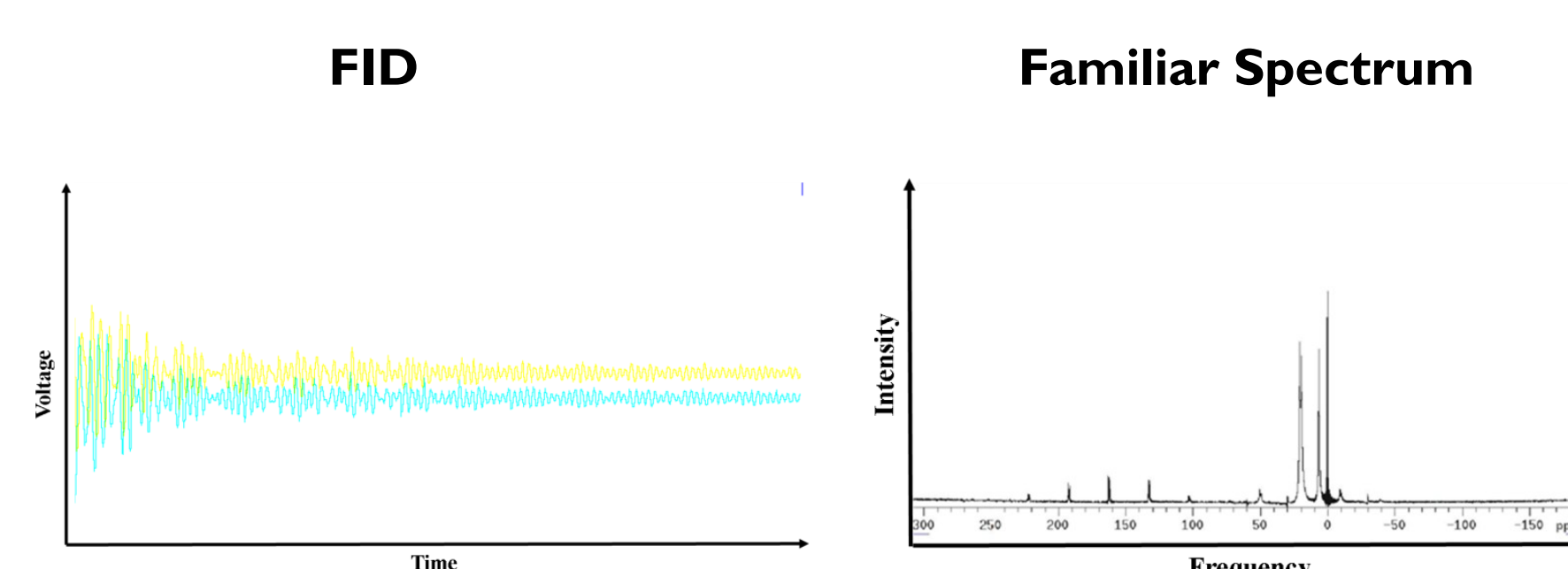
Magic Angle Spinning



Graphical Tuning

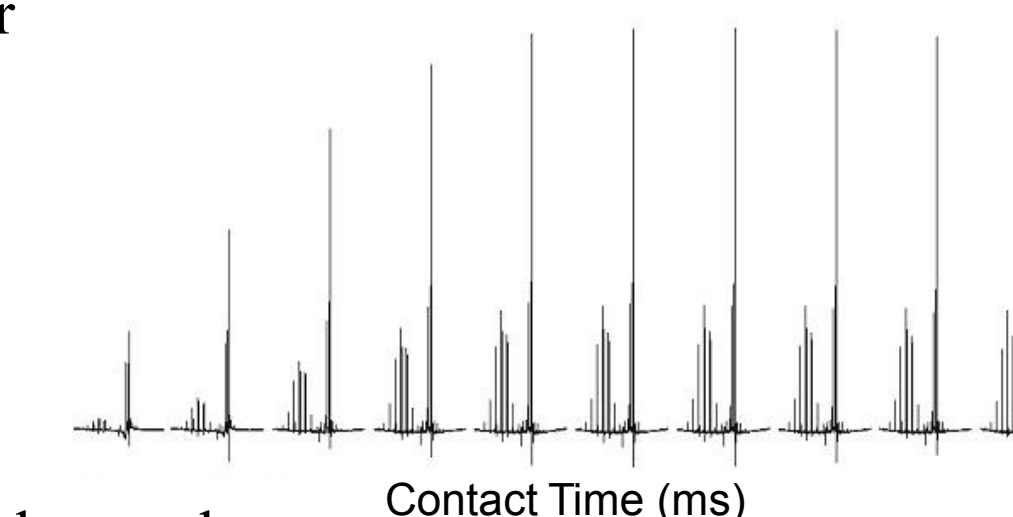


Fourier Transform



Parameter Arrays

- Experiment repeated with only one parameter changed
- Spectra plotted horizontally
- Intensity changes observed

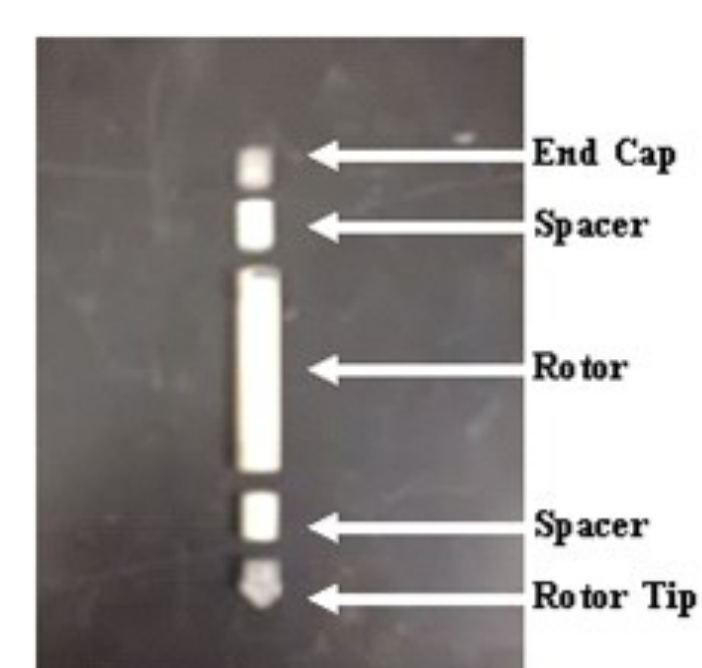


Conclusion

- Solid-State NMR is becoming mainstream
 - Industry
 - Undergraduate level
- Research interests
- Documentation

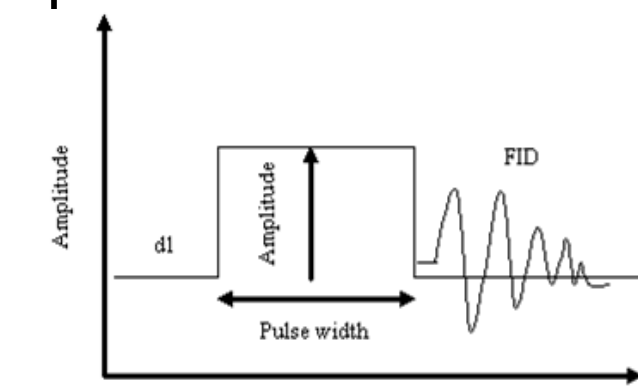
Rotor Preparation

- Key aspect of proper MAS
- Rotor holds sample and must be able to withstand high spinning rate
 - Ceramic zirconia
 - Rotor explosions



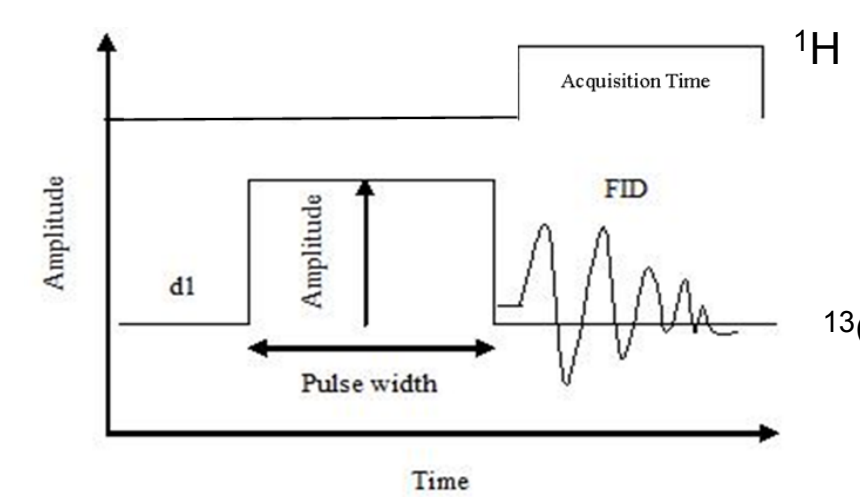
Pulse Sequences

- Describes what the spectrometer "does" during an experiment
 - Explains the RF pulses by indicating amplitude, phase, and duration
 - Shows delays
 - Shows acquisition (receiver)
- Single Pulse Excitation (SPE) pulse sequence
 - Most basic pulse sequence

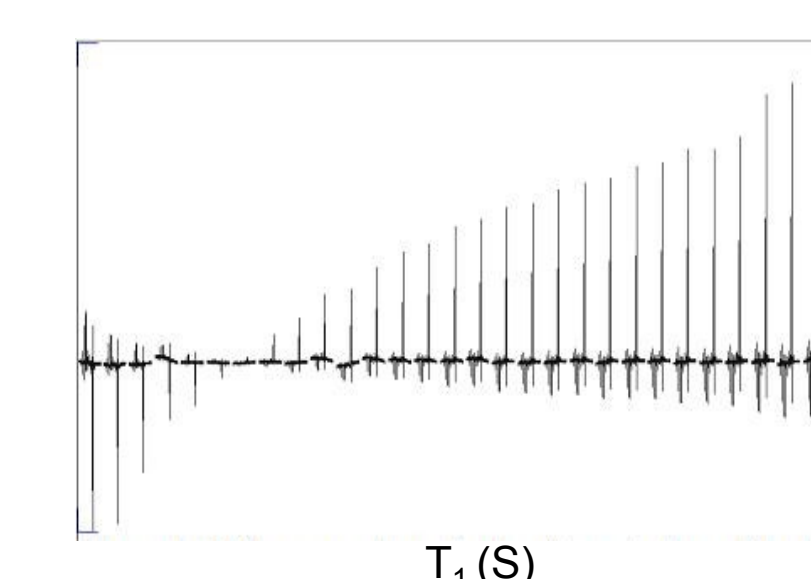


Dual Channel SPE

- Allows for decoupling while acquiring
- Double resonant experiment
- Coil is tuned to two frequencies simultaneously
 - ^1H -400MHz
 - ^{13}C -100MHz



Parameter Arrays



Acknowledgements

I would like to thank Dr. Iuliucci for the guidance and support that he has provided for the last two years. I would also like to thank the Magellan Project and the Howard Hughes Medical Institute for providing the funding needed to pursue the various projects that I have undertaken during my time at Washington & Jefferson College.

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