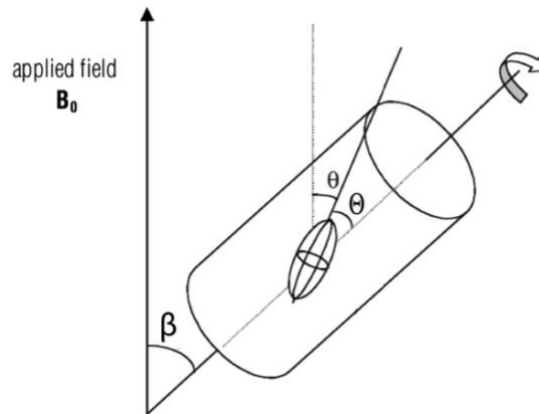


## Jigsaw 5D

### Solid-State NMR

1. [Week 4 Slides 13–43] The diagram shows the schematic sample setup for a magic-angle spinning experiment.



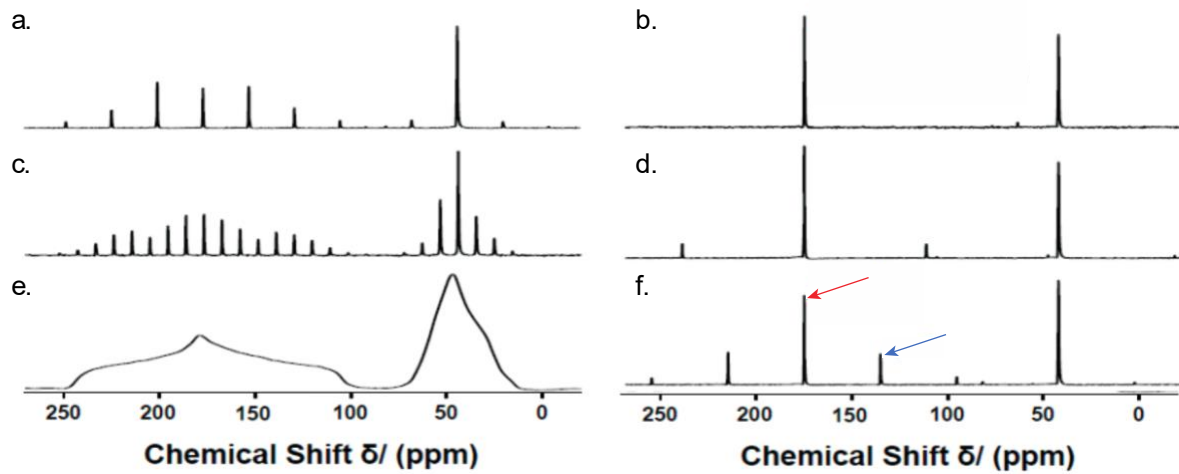
- a. Define the angles  $\beta$ ,  $\theta$  and  $\Theta$ .
- b. Name three interactions which are averaged or partially averaged by sample spinning.
- c. For a nucleus with a chemical shielding tensor with axial symmetry, we can write the chemical shift as the sum of isotropic and anisotropic terms:

$$\delta = \delta_{iso} + (\delta_{zz} - \delta_{iso}) \left( \frac{3 \cos^2 \beta - 1}{2} \right)$$

For which angle  $\beta$  is the anisotropic part 0?

- d. What is the effect of spinning the sample around an axis oriented at  $\beta = 90^\circ$ ?

2. Here are six solid-state NMR spectra of the same sample recorded at different MAS spinning rates: 0kHz (static), 1kHz, 3kHz, 5kHz, 8kHz and 14kHz.



- i. Which spectrum corresponds to each spinning rate?
- ii. The red arrow indicates the isotropic shift, but what does the second arrow correspond to?
- iii. What does the spacing between the peaks in part (ii) depend on?

3. [From past exam] Consider the principal values for the following  $^{13}\text{C}$  chemical shift anisotropy (CSA) tensors:

- a.  $\delta_{11} = \delta_{22} = \delta_{33} = 50$  ppm
- b.  $\delta_{11} = 30$  ppm,  $\delta_{22} = 10$  ppm,  $\delta_{33} = 0$  ppm

Sketch the powder pattern of the  $^{13}\text{C}$  spectra for **both static and Magic Angle Spinning (MAS)** conditions for each CSA tensor. Of the two static spectra, which has a larger value of span ( $\Omega$ )?