CH-314 Structural Analysis
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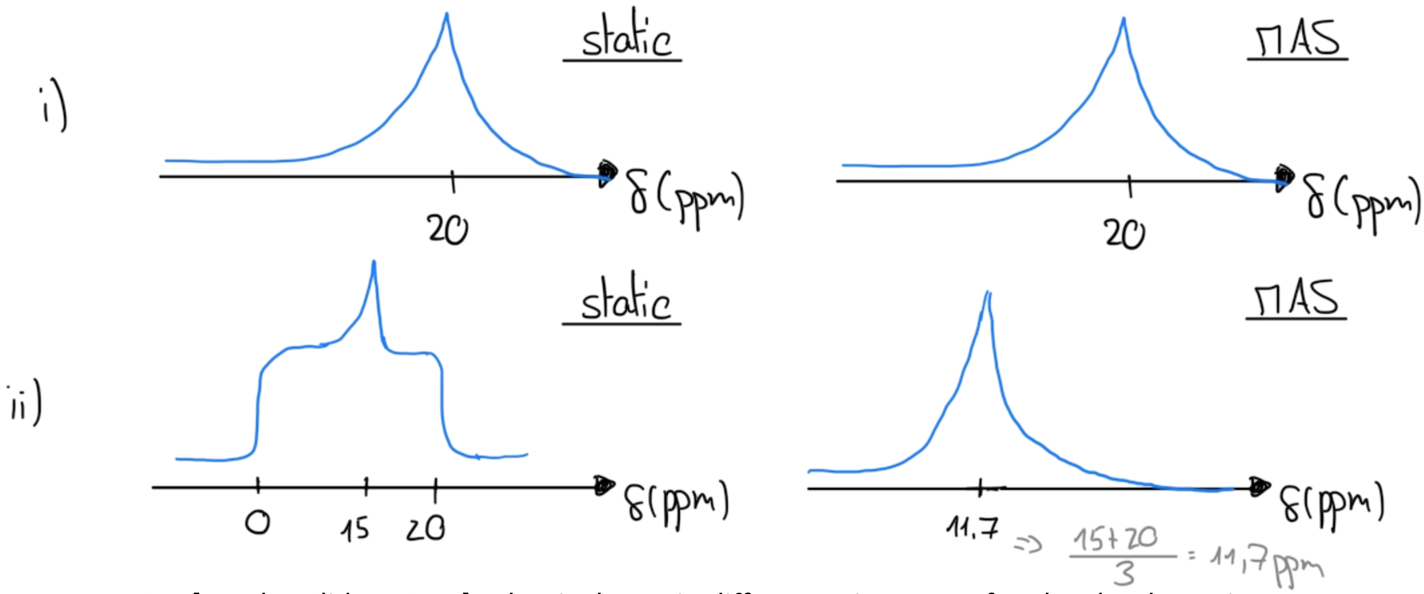
Jigsaw 5C

1. [From Past Exam] [Week 4 Slides 25-41] Consider the principal values for the following ^{13}C chemical shift anisotropy (CSA) tensors:

   (i) $\delta_{11} = \delta_{22} = \delta_{33} = 20$ ppm
   (ii) $\delta_{11} = 20$ ppm, $\delta_{22} = 15$ ppm, $\delta_{33} = 0$ ppm

   Sketch the powder pattern of the ^{13}C spectra for both static and Magic Angle Spinning (MAS) conditions for each CSA tensor. **2/2**



2. [Week 4 Slides 43-44] What is the main difference, in terms of molecular dynamics, between a liquid and a solid sample? **2/2**

   In liquids, molecules exhibit rapid isotropic motion.
   In solids, molecular motion is restricted. **...but there is still tumbling while spinning but rotational molecular motion is much lower than 1/\Omega**

3. [From Past Exam] [Hore Section 3.3] The ^{93}Nb spectrum of $[NbOF_4]^-$ is a quintet. The ^{19}F spectrum has ten equally spaced lines with the same intensity. What is the nuclear spin of ^{93}Nb? **2/2**

   For a nucleus of spin I, the number of lines is given by $2I + 1$.
   $\Rightarrow 2I + 1 = 10 \Leftrightarrow I = 4{,}5$, so the nuclear spin of ^{93}Nb is 4,5.

To be more general, for a spin S, will find 2In+1 lines due to coupling with neighbour with spin I; remember that n is the number of neighbours! (in this case it is 1, true)

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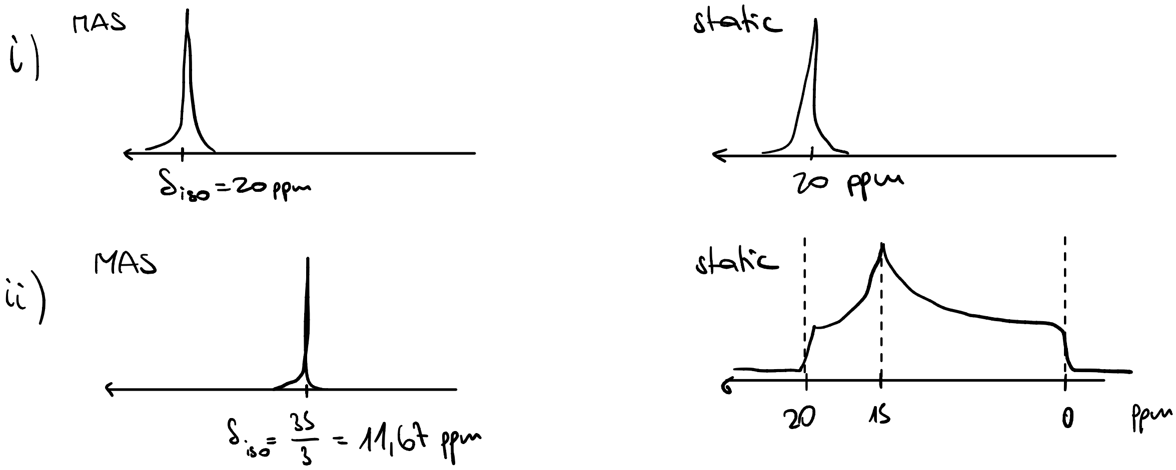

$$\delta_{iso} = \frac{\delta_{11} + \delta_{22} + \delta_{33}}{3}$$

2. [Week 4 Slides 43-44] What is the main difference, in terms of molecular dynamics, between a liquid and a solid sample? 2/2

In liquid we have more tumbling motion which average the three principal components of the chemical shift tensor to the same value and thus a single peak with shift δ_{iso} (isotropic) is observed.

In solid there is no tumbling motion, therefore each crystallite appears at a different shift depending on its orientation relative to the magnetic field. There is still tumbling while spinning but rotational molecular motion is much lower than 1/\Omega

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Nb of lines = multiplicity

Spectre ^{93}Nb:

Since the complex $[NbOF_4]^-$ is a quintet and we have 4 ^{19}F then the spin of ^{19}F is $\frac{1}{2}$.

Spectre ^{19}F:

we have one signal with multiplicity ten.
The multiplicity is $2I + 1$, with I the nuclear spin.

So: $10 = 2I_{^{93}Nb} + 1 \Rightarrow I_{^{93}Nb} = \frac{9}{2}$