WESTFÄLISCHE
WILHELMS-UNIVERSITÄT
MÜNSTER

INSTITUT FÜR PHYSIKALISCHE CHEMIE

Studiengang MASTER OF SCIENCE Chemie

MODUL Spektroskopie und Struktur der Materie

SIMPSON – SOLID STATE NMR I

General Remarks:

The following tasks will provide basic knowledge on the function and application of the SIMPSON simulation software. Subsequent to this course a protocol has to be prepared, typically including one or a few graphics for each task and a short explanation for the observed spectra/curves. While the practical tasks should be completed individually, protocols can be prepared in teams of two students. Detailed information for the protocol is given in brackets [ ].

1. STATIC AND MAS NMR CONDITIONS

Two SIMPSON input files are provided: ***static.in*** and ***mas.in***. In addition, three more crystal-files are also provided. All files should be placed in a single folder for this exercise. The aim of this exercise is to understand the anisotropy of the chemical shift influence, the resulting static line shape originating from different orientations of single crystals, and the Magic Angle Spinning (MAS). Make sure to save the results of each task (change the input file name)!

a) In the ***static.in*** change the `spinsys` section to represent a ^{19}F nucleus with a chemical shift anisotropy of ca. +10 ppm and an asymmetry parameter of ca. 0.42. Simulate the spectrum of a single crystallite for different sets of Euler Angles for the CSA interaction (0 0 0), (0 90 0), and (90 90 0). Make sure to use the crystal file 'alpha0beta0'. Save the spectra! [(Stack-)plot the spectra and explain the differences]

b) Now simulate a powder spectrum containing multiple crystallites by using a different crystal file ('rep10', 'rep30', 'rep100', 'rep2000', 'zcw75024'). How many crystallites are necessary for sufficient powder averaging? [Prepare a meaningful graphic including 3-5 spectra, which crystal file would you prefer ?]

c) Modify the parameters of the CSA, e.g. change the sign of δ_σ and vary the asymmetry parameter η_σ between 0.0 and 1.0. Observe the influence on the spectra. [Plot selected spectra that are suitable to explain the influence of each parameter].

d) Simulate the effect of MAS on a CSA spectrum by using the ***mas.in*** input file (you might need to adjust some parameters). Start with a MAS frequency of 1.0 kHz and a crystal file of 'rep678'. Vary the number of 'gamma_angles' between 1 and ca. 30 until the spectrum is not changing (i.e. convergence of the gamma_angles). [Plot spectra for different gamma angles and explain the differences, also compared to the static case]

e) Next, vary the MAS frequency in ca. 1 kHz steps until the CSA is averaged completely. [Prepare a meaningful graphic including 3-5 spectra, which rate do you prefer ?]

Bonus: If you are interested, you can simulate off-angle MAS by varying the parameter 'rotor_angle'. By omitting or commenting out 'rotor_angle', a perfectly set magic angle is assumed.