First substance for NMR!?

How much substance do I need?

For ¹H-spectra:

5-10 mg for quick measurements at the 200 MHz-spectrometer.

Less (down to 1 mg) is acceptable for protons, the measurement will just take more time (for example 15-20 min instead of 2 min at 500 MHz).

For ¹³C-spectra:

The amount of substance and amount of carbon atoms in the molecule have great influence on the time of measurement.

~ 0.5 mg per expected carbon signal is enough (500 MHz)

Note: Two-dimensional spectra (HSQC, HMBC) are much more sensitive for small amounts of substance than one-dimensional spectra (¹³C-CPD or ¹³C-DEPT)

How much solvent should I use?

The solvent level should be exactly 4 cm (+/-2 mm).

Higher levels give shimming problems which might result in an asymmetry of all signals; lower levels can't be measured at all.

The substance should be dissolved completely, unsolved particles disturb the measurement. (For substances of low solubility change the solvent and/or use less amount of substance, maybe centrifuge)

How do I fill out the form?

READABLE and COMPLETELY!

The structure must be comprehensible.

Information about the quantity is obligatory! Weighed on a scale or at least estimated!! Your telephone number is needed in case of queries.

After checking the sample purity in a 1D-¹H-NMR-spektrum on the 200 MHz instrument, if you need further measurements, fill in the number of the already measured spectrum (e.g. Prak-OCG-160525-15). Likewise if the substance has already been measured at 500 or 600 MHz (e.g. I511Meye_1234).

What kind of spectra do I need?

Always one spectrum of 200 MHz to check the purity.

If you need the full characterization:

COSY is only needed, if there are more than three coupling protons.

HSQC shows the direct coupling of ${}^{1}\text{H}{}^{-13}\text{C}$ (bonded!).

HSQC-DEPT version shows negative (red) signals for CH₂-groups

HMBC is useful to characterize quarternary carbons which couple to a proton via 3 bonds, and as well, if the amount of substance is too small to achieve a one-dimensional ¹³C-Spectrum.

NOESY is necessary in case you want to observe couplings through space. (Only needed in special cases)

¹⁹F measurement is as fast as proton measurement.

¹¹Bor, ²⁹Si, ³¹P, ⁵¹V, ⁹¹Zr, ¹¹⁹Sn are standard experiments, for other nuclei just inquire.

Feel free to ask, if you are unsure about anything! ©