



Sample preparation for NMR

In NMR the adequate sample preparation is of great importance for the quality of the spectra, the sample should not contain any solvents or impurities which can interfere with the signals that are acquired.

For our equipment Bruker Ascend 400 which works at 400MHz the adequate sample amount is 1-5 mg, for spectra of proton 1D and Fluorine, while for the spectrum of ^{13}C and 2D spectra the right sample amount is approximately 20 mg.

All used solvents must be **deuterated**. In this way we can get rid off of the solvent signals in the proton spectra. At the Centre we have available **Dimethylsulfoxide-d₆** (99.80%)+ 0.03% (v/v) TMS, **Deuterium oxide** (99.90%), **Chloroform-d** (99.80%)+ 0.03% (v/v) TMS. In case the sample is soluble in another solvent, it has to be prepared beforehand by the applicant.

The adequate sample volume is about **0.7 mL** (approximate height of 5 cm). Sample volumes smaller than 0.6 mL could lead to problems of homogeneity. The use of higher volumes (than 0.7 mL) is not recommended because can derive in temperature gradients in the sample.

The used probes should be of high quality and appropriate for the equipment. For our equipment at 400MHz commercial probes **Wilmad 507-99-7** are recommended. The samples will be prepared by the technician and should be delivered on **Tuesday from 09:00 to 13:00h**. All samples should be delivered together with the application form.

The results of the analysis will be sent by e-mail to the applicant. The program to process all spectra can be found on the web page of Bruker:
<https://www.bruker.com/service/support-upgrades/software-downloads/nmr.html>
(Free Academia License).