SPECTROPOLE
Centre d'analyses chimiques

SAMPLE PREPARATION

I – NMR Tubes

The NMR tubes must satisfy certain quality criteria necessary for a good analysis (for further details contact the SPECTROPOLE manager). Before being used, the tubes must be clean and dry with no breaks or cracks (proton sensitivity is such that any impurity will be detected, which could affect the analysis and/or interpretation of the data). The minimum length of these tubes must be 170 mm so that the arm can grasp the tube correctly and not by the cap.

II – Tube cleaning

After analysis, the tubes must be washed as quickly as possible with the solvent used to solubilise the product under study. After several rinses, the tube is dried under vacuum.

III – Tube Filling

Resolution depending on magnetic field homogeneity, this parameter is automatically fine-tuned for each sample. Under the standard analysis conditions pre-programmed on the instrument, the homogeneity of the field has been calibrated using reference tubes with a constant filling height of 4 cm, *i.e.* 600 µl of solvent. In order to obtain optimum measurement conditions, you must respect this height of solvent in your tube.

IV – Sample Concentration

The sensitivity of an NMR experiment depends on the nuclei studied and the concentration of your product. Under the standard conditions pre-programmed on the instrument, a concentration of 0.6M is required (in 600 µl of liquid). For example:

RMN^{1}H and RMN ^{31}P ~ 5 mg

RMN^{13}C during the day ~ 30 mg

RMN^{13}C night ~ 10 mg

V - Suspended solid

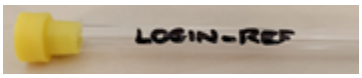
A solid product does not have the same magnetic susceptibility as a product in liquid phase. It is therefore necessary, when preparing your tubes, to filter your solutions in order to eliminate all solid particles.

VI - Choice of deuterated solvent

The choice of deuterated solvent depends mainly on the solubility of your product at room temperature. If you have several options, choose the solvent whose peaks are unlikely to interfere with the peaks of your product. Solvents with labile D (CD_3OD, D_2O) are very useful for checking the presence of an acid proton in your molecule (OH, NH). If you want to work at a particular temperature, remember to choose a solvent whose boiling point (high temperature) or solidification point (low temperature) is at least 10° C higher or lower than the maximum temperature at which you want to work (in all cases, talk to the person in charge beforehand).

VII – Tubes labeling

No glued labels or paper will be allowed on the tubes. You will need to label your tubes by writing on the TOP of the tube with a permanent black marker, your login and the sample reference.



VIII – BACS passer, spinner

Please take care of the sample changers and turbines by handling them with CLEAN hands, without gloves.

DO NOT, UNDER ANY CIRCUMSTANCES, RELY ON THE SAMPLE CHANGER.

Once inserted into a spinner, the tube will be calibrated by putting it in the calibrator attached to the wall and pushing it all the way to the bottom without forcing.