

Leaflet for sample preparation

Solvent and standard:

- Please use no deliverable mixtures with more than 0.03 volume percent of TMS.
- Consider that some solvents are hygroscopic.
- For the addition of TMS, we suggest the following procedure: The gas room in the TMS flask is grasped with a Pasteur pipette which is then flushed five times with air. Afterwards the residual TMS in the pipette gas room is bubbled through the prepared NMR-solution.

Tubes:

- We use only NMR tubes of quality 507 PP and 5 mm outer diameter. This quality was recommended also by the manufacturer of the NMR devices, in order to prevent damages in the probes which we use.
- The length of the tube must be 18 cm minimum (shorter ones are refused, because of the risk of tube burst).
- Partly broken tubes ensure no secure fit of the caps and must be cut off planar by the glassblower before use.
- For light-sensitive substances we borrow dark colored tubes.

Closing of the tubes:

- The tubes must be provided with tight caps, may be you should wrap a thin layer of Parafilm around the tube, in addition.

Contents:

- Our devices are adjusted to 5 cm height of filling (ca. 0.8 ml of solvent). Please be as precise as possible! Deviations from this rule (particularly to the low side) mean much worse resolution in the spectra.

Concentrations:

- ^1H : 0.025 mmol
- ^{13}C : 0.1 mmol
- ^{31}P : 0.1 mmol
- ^{19}F : 0.1 mmol
- These amounts (in 0.8 ml) represent our experience to obtain reasonable spectra with pure substances in routine measurements with standard parameters at the AVANCE 300. Deviations to smaller concentrations results in a dramatic loss of signal/noise ratio.

Quality of the solution:

- The sample should not contain suspended material (i. e. fluff), because this strongly reduces the quality of the spectra.
- Consider also that weakly soluble substances can precipitate again.

Filtration:

- In case of problem we recommend filtration over cotton (Strongly wrap some cotton around the tip of a Pasteur pipette and suck the solution; strip off the cotton and fill the solution into the NMR tube)
- In case of very fine precipitation we recommend a filtration with so-called membrane filter.

Cleanliness:

- Before delivery, the tubes must be wiped clean. Contaminated tubes will be refused in order to protect the staff.

Labeling:

- The samples must be provided with the name of the customer and a name of the substance (agreeing with the one on the order paper!).
- For this purpose, please use easily removeable paper labels and do not use adhesive labels.