NMR_MAGRITEK

1. Preparing and loading a sample:

a) Put the sample in the NMR tube (Manual, page 15).

The sample volume is 300 – 500 μL (only up to the black marker line, **Fig. 1A**). Remove all solid particles. Solid particles can distort the magnetic field homogeneity because the magnetic susceptibility of a particle is different from that of the solution. A sample containing suspended particles thus has a field homogeneity distortion around every single particle. This causes broad lines and indistinct spectra that cannot be corrected.

b) Put the tube in a spinner, **Fig. 1B** (Manual, page 16).

c) Check sample level **Fig. 1A** (Manual, page 16).

d) Put tube and spinner into Spinsolve spectrometer, **Fig. 1C** (page 19).

NOTES:

- Use an Eppendorf tube or similar for sample preparation.
- You may use either deuterated or non-deuterated solvents. Non-deuterated solvents will give significant solvent residue peaks. This may overlap sample peaks in the spectrum. The sample concentration should be around 50 mM for this device.
- For 250 mM concentration, use a STANDARTSCAN mode (1 min). For 50 mM or lower concentration, use a POWERSCAN (10 min) mode.
- The sample must be soluble in a solvent. To prepare a homogenous solution, use a sonic bath, a heating gun (pay attention to the solvent's boiling point), or a syringe filter.
- Solvents available in the Chemical laboratory are chloroform, methanol, and benzene, located on the shelf under the fume hood. Work with solvent at room temperature (RT).
- During NMR measurement, it is forbidden to use vibration devices close to the NMR spectrometer (less than 1.5 m).

Okomentoval(a): [ZJ(1]: Nejsem si jistý zkratkou...

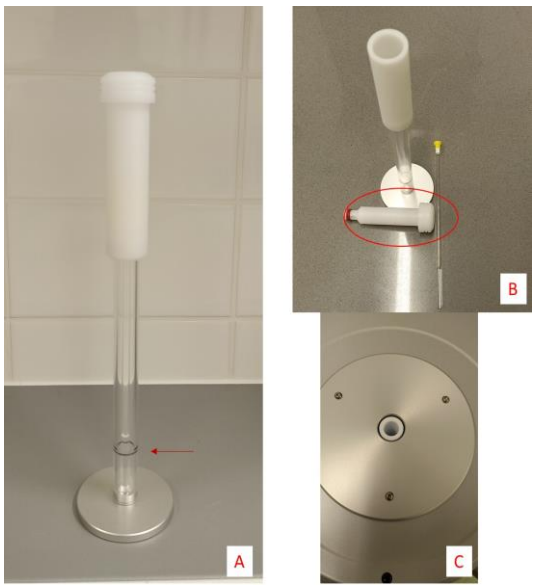

Fig. 1: (A) Sample level. (B) Spinner (labeled) and holder. Magnetic stirrer, centrifuge, and other magnetic. (C) The measurement cell.

2. Measurement:

a) The front pillar's light indicator should always be green (Fig. 2A). If not, contact the responsible person.
b) The instrument is in standby mode for the optimization of a permanent magnet. Push the STOP box (Fig. 2B).
c) Leave the reference sample in the probe, and press the QUICKSHIM box (Fig. 2C). Wait until the end of shimming. Now the instrument is ready for measurement.
d) Exchange reference sample to the measured one.
e) Select a protocol at the top of the screen.
f) Measured dates can be evaluated in the Spinsolve program (panels below the graph), exported to NOVA, or saved data.
g) For more detail, use Manual or "Help" in the Spinsolve program. Measured data are automatically saved in the PC-Windows folder: (C): PROJECTS /DATA / 2023 – 01 (month) – 01 (date). Data save here.
h) Saved data can be sent or saved on SharePoint (do not use a USB key).

NOTES:

- In the booking system, write the information about measurement in the Log Book for the NMR_MAGRITEK instrument.

Essential points for obtaining good results

- Well-prepared sample (homogenous). Remove all solid particles.
- Volume – better a little bit upper the black marker line in the sample holder (Fig. 1A) than lower.
- Correct position of an NMR tube in the sample holder.

3. Finnish of measurement:

a) Put the reference sample into the probe. Switch on the Standby mode.
b) Remove the sample from the NMR tube, or it will be trashed. Put the NMR tube into the beaker labeled "Dirty BET + NMR".

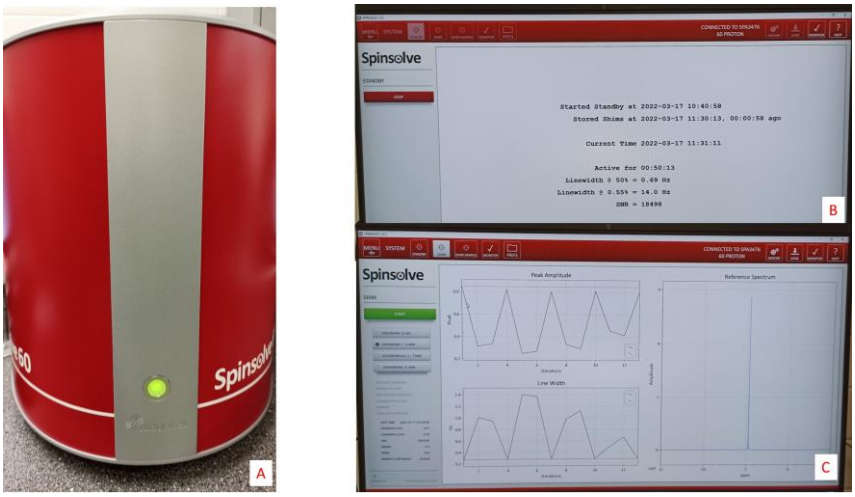

Fig. 2: (A) Ready instrument. (B): STOP standby mode. (C): Start shimming the reference sample.