

General guidelines and rules of the OC NMR department (as of 25.07.2023)

Service NMR:

- ☐ Requests for measurements should be done through submission of a sample with a request slip
- ☐ Contact with NMR personnel preferred over the phone or by email

Self-service operation of the Open-Access spectrometer (after instruction only):

- ☐ The Open-Access Spectrometer is available to researchers of the Organic Chemistry Institute
- ☐ For instruction, please contact Timon Reckmann (timon.reckmann@wwu.de , Tel. 39773)
 - 1D NMR: ^1H , ^{19}F , ^{11}B , and ^{31}P NMR spectra (only concentrated samples!)
 - **Maximum of three samples per person at any one time**

400 MHz NMR services available on request:

- Urgent ^1H and ^{19}F NMR measurements (will be measured in priority)
(e.g. if there are too many samples in the queue of the access spectrometer!)
- ^{13}C , heteronuclear, and specialized ^{19}F NMR measurements
- Serial measurements

High-field NMR:

- Specialized 1D/2D NMR measurements – see the request slip for details
- Other specialized techniques available on request
- ☐ As a rule, only accepted after initial confirmation of purity through ^1H and ^{19}F NMR on the 400 MHz spectrometer
- ☐ **A list of all $\delta(^{19}\text{F})$ values must be given on the request slip for samples containing fluorine**

Labelling of samples:

- ☐ **No letters or symbols** should be used to label samples beyond those of the user's three-letter abbreviation, e.g.:
mue 591-1 (= **user identifier** & **reaction/sample number**)

Request slips and sample labels:

- ☐ Template should be downloaded, filled out, and printed in advance (Don't plot the paper double-sided!)
(<https://www.uni-muenster.de/Chemie.oc/en/nmr/nmr-service.html>)
- ☐ The solvent, amount of analyte, reaction number and requested experiments should be filled out by hand in legible handwriting
- ☐ One form for several samples only with the same solvent and with identical measurement requirements

Sample preparation:

- ☐ The exterior of NMR tubes must be absolutely clean
- ☐ NMR tubes must be **18–21.5 cm long**; broken or damaged NMR tubes and caps are forbidden
- ☐ Tubes should be filled to 5 cm with samples, in deuterated solvents only (**no mixtures!**)

Special case: serial measurements of x samples (where $x > 3$):

- ☐ Samples will be numbered **1 through x**
- ☐ **Only possible for samples in the same solvent and with identical measurement requirements**
- ☐ Instead of attaching a sample label, the caps should be marked 1 through x and placed in the provided sample stands (to the right of the department entrance) with one request slip per series

Sample submission and return:

- ☐ In front of the department entrance (observing all signs)

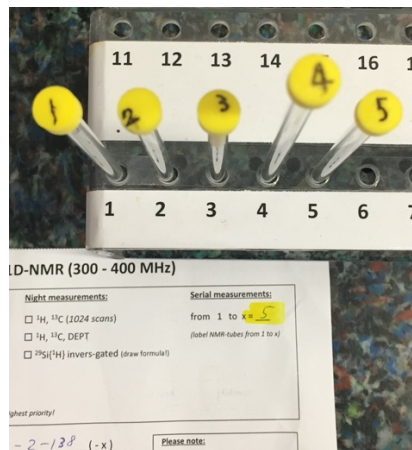
Download of raw data:

- ☐ Data from all spectrometers can be downloaded onto the user's PC immediately after measurement with the program "mora the explorer".

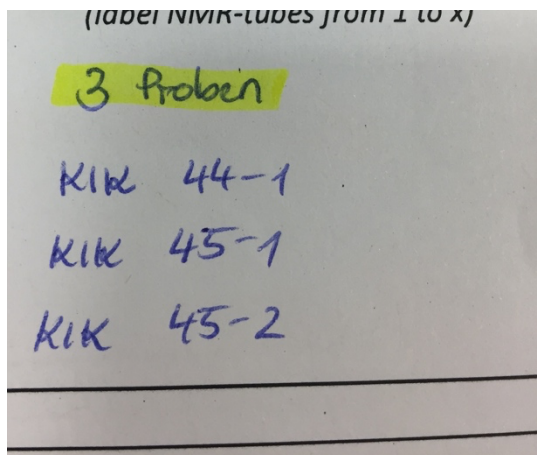
Examples:

Several samples with the **same solvent** and **identical measurement requirements:**

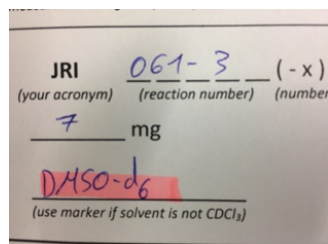
Serial measurements:
(only for more than 3 samples!)



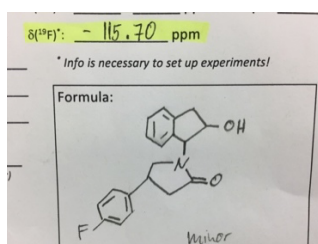
Up to three samples:
(One form, three labels)



For solvents other than CDCl_3 :



Highfield-NMR of Fluor containing compounds
list of all $\delta(^{19}\text{F})$ is required:



Acknowledgment:

We thank Matt Milner for kind translation and especially for providing the program "mora the explorer"!

Final note:

- ☐ Samples that do not conform to the guidelines 100% will be returned unmeasured.

Operation of the Open-Access NMR spectrometer (as of 27.04.2023)

- ☐ **Only trained persons may operate the open-access spectrometer.** Instruction can be obtained from an employee of the NMR department or from the person responsible within your research group.

Safety around a strong magnetic field:

- ☐ **No access for:**
 - persons with pacemakers, diabetes pumps, or metallic implants (**risk to life!**)
 - employees, researchers, and students who have not received instruction in the use of the device
 - anyone wearing a lab coat
- ☐ **Ferromagnetic objects**, including **phones, keys, coins, and credit cards**, must be removed from the person and laid on the table directly by the door (**risk of injury** if objects are pulled to the magnet!)
- ☐ The rooms must be kept completely clean!
 - Smashed NMR tubes should be disposed of appropriately; any contaminated surfaces and components must be thoroughly cleaned with isopropanol! Inform an employee of the NMR department!

Sample preparation and labelling:

- ☐ As an exception to the rules on the first page, a request slip does not need to be filled out
- ☐ **Only pure deuterated solvents!** (also, no mixtures of deuterated and non-deuterated solvents)
- ☐ A **maximum of three samples** are allowed per person simultaneously; otherwise use the normal NMR service e.g. for serial measurements

Operation of the sample autochanger and queuing of measurements:

1. Take your sample tube, look for the **next free accessible position** of the sample changer and note this number on your sample label
2. Hold a spinner by the upper edge and position the NMR tube with the help of the gauge
3. Wipe the NMR tube with a Kim wipe (Do not shift the position of the NMR tube in the spinner!)
4. Put the spinner in the **right position** (cf. 1.) when the autochanger is **not moving**; check if you've noted the position occupied on the sample's label and take your label(s) to the computer
5. On the computer, input the position number, solvent, and required experiments
Title input is exclusively in the format: **group abbrev.** **user abbrev.** **reaction/sample number**
e.g.: **stu** **mue** **591-1**
the reaction number **may not contain letters or symbols** (only hyphens are allowed)!
6. Place your sample labels with the others of your group in ascending order
7. Take all labels that belong to your group to the computer, select all labels of the completed measurements and delete completed measurements from the autosampler queue
8. Remove sample tubes from completed measurements that belong to your group, attach the respective labels and put the sample tubes to the small table provided for sample return (on the left side to the NMR entrance)

Troubleshooting:

- ☐ General questions should be addressed to the responsible person within your research group.
- ☐ Problems with the device should be immediately reported to an employee of the NMR department:
Tel. Timon: 39773, Ingo: 36509, Klaus: 39776, klaube@wwwu.de
- ☐ I confirm, I have read carefully and understood these guidelines.