plateforme RMN	**Users' Charter NMR Facility**	UNIVERSITÉ DE LORRAINE

PREAMBLE

This charter sets out the obligations of users of the CPM (Molecular Physics & Chemistry) Science Cluster's NMR facility.
It is understood that by adhering to this charter, the user undertakes to respect or ensure respect for all the rules below.
The commitments are materialized by the signature of the present document by each person concerned, thus attesting that he or she has read of the said charter.

1. Presentation of the NMR facility

The objective of the CPM Science Cluster's NMR facility is to make the NMR tool accessible to the entire scientific and industrial community in Lorraine. To this end, it has a wide range of spectrometers (see Appendix 1) from 100 to 600 MHz covering all NMR applications (liquid, solid and imaging).

The equipment is installed on the campus of the Faculty of Science in Vandoeuvre-lès-Nancy, and one device is also available at the Metz technopole. Two instruments at the Nancy site, described below, are freely available after training by a person in charge of the NMR facility.

The two open access devices are :

- « 400 direct » : A Bruker Avance III 400 MHz spectrometer equipped with a direct detection probe (BBFO 5mm probe: double resonance, 1H, 2H and X nuclei, direct detection of heteronuclei (including 19F), Z gradients 50 G/cm, automatic tuning of the probe), an air cooler BCU-05 and a 60-position sample changer.

  The instrument is used in the form of slots reserved for each laboratory.
  Optimized detection of carbon-13.

- « 400 inverse » : A Bruker Avance III 400 MHz spectrometer equipped with an inverse detection probe (BBI 5mm probe: dual resonance, 1H, 2H and X nuclei, inverse detection of heteronuclei, Z 50 G/cm gradients, automatic probe tuning) and a 60-position sample changer.

  This apparatus is used in free access. Long experiments (especially two-dimensional heteronuclear experiments, >15min) are automatically started at night (6pm-8am).
  Optimized proton detection.

The NMR facility's staff manages the facility's entire instrumental pool, analyses and develops methodologies. In particular, they ensure the performance and maintenance of the equipment, the planning of occupancy times and the reservation of equipment. They also carry out specific experiments (solid state NMR, diffusion, temperature variation, etc.).

2. Operation and access

The NMR facility equipment is accessible to all users, either through requests for specific projects (manipulations carried out by NMR facility staff and/or advanced users) or by making the two 400MHz devices available.

For specific project requests, users should contact the responsible persons in order to study the feasibility and, if necessary, to establish a schedule of experiments in agreement with the NMR facility managers.

The user must be trained to use the two 400MHz instruments in free access (described in chapter 1). He then becomes autonomous to carry out the experiments for which he has been trained (appendix 4), and commits himself to respect the procedures of use as well as the schedules (appendix 2). In case of doubt, ask the person in charge of the equipment. Concerning the periods "on reservation", the requests must be made via the website: https://resa-ijb.univ-lorraine.fr and are submitted to the NMR facility managers who manage the schedules. In order to satisfy users' requests as much as possible, it is imperative that reservation requests be made as soon as possible.

Opening hours of the service: Monday to Friday from 8am to 6pm. Outside these times, a request must be made to the NMR facility manager.

The NMR facility is closed when the university is closed.

3. Hygiene and safety

Each user is required to respect the rules of hygiene and safety and good laboratory practice applied in public laboratories.

It is forbidden to eat and drink on the premises.

Due to the presence of intense magnetic fields, access to the equipment is strictly forbidden to persons with medical contraindications (pacemakers, ferromagnetic surgical implants, implanted electronic devices, etc.). It is absolutely necessary for each permanent staff member to check with his or her trainees about this particularity. Furthermore, it is forbidden to enter the 5 Gauss zone of each device (marked by a yellow/black line on the floor) with any ferromagnetic or magnetic field-sensitive object (keys, mobile phone, magnetic card, metal objects, etc.).

The user is responsible for being aware of the chemical and/or biological risks associated with the samples he/she brings onto the site. They must inform the NMR facility staff of the specific risks and safety measures associated with the handling of their samples.

The oxygen content of the spectrometer rooms is measured continuously. In the event of an unacceptable level, a visual and audible alarm will indicate the evacuation of the room. This evacuation is compulsory and must be carried out as quickly as possible in a calm atmosphere and via the exits indicated by the signs.

The doors of the spectrometer rooms must be kept closed so that the temperature of the rooms remains constant.

4. NMR facility commitment

As part of the quality approach undertaken by the NMR facility, it is committed to satisfying the needs of its users by updating its knowledge, perfecting its equipment and continuously improving its organization, in compliance with regulatory and legal requirements.

Those in charge of the NMR facility undertake to study the feasibility of the requests and inform the future user of their suitability for the NMR facility's instruments and skills.

Within the framework of the provision of equipment, users retain ownership of their results. The NMR facility undertakes not to use these results without the explicit permission of their owner.

The NMR facility is committed to its users to:
- provide quality services.

- manage the schedules and flow of requests to use the spectrometers according to human and material availability.
- train the user so that he/she is autonomous on certain equipment made available (see chapter 2).
- inform the user of any malfunction that could affect his project.
- Respond to any complaints from users.

5. User engagement

5.1 Samples

All users undertake to provide samples prepared in accordance with the NMR facility's recommendations and the established specifications, in particular with regard to solvent volumes, maximum or minimum product mass and tube cleanliness (Appendix 3). The NMR facility is entitled to refuse samples delivered in poor conditions.
He also undertakes to take back the samples after analysis, or exceptionally and with the agreement of the NMR facility managers, to indicate the waste disposal procedure.

5.2 Use of computer equipment

The user undertakes to use the NMR facility's computers only to carry out his/her experiments. This use must comply with the University of Lorraine's IT charter. The installation of software without the agreement of the managers is prohibited. After use, the user must log out of his session. In the case of the use of the sample changer, the session may remain open for the duration of the slot allocated to the laboratory but must be closed at the end of the slot.
The user is the owner of his data and is responsible for its backup and archiving. The NMR department regularly deletes data in order to conserve space on the hard drives.

5.3 Hardware

All users must comply with the procedures for using and, where applicable (advanced users only), switching on and off the equipment. These procedures are established and can only be modified by the NMR facility staff according to the specifications of the hardware suppliers.
No modification or adaptation must be made to the various equipment, even temporary, without the consent of the NMR facility manager.
The user undertakes to report any malfunctioning to the managers.

5.4 Responsibility

The instruments are made available to the users who are obliged to take care of them, to respect the rules of the art and the instructions for use, to respect the rules of hygiene and safety in accordance with good laboratory practice.
During their presence on the NMR facility, users remain under the responsibility of the director of their laboratory and of the organization to which they belong. In the event that a user is directly responsible for damage to equipment, his/her team/laboratory must take charge of the necessary repairs or replacements.

5.5 Respect for accessibility

All users must respect the NMR facility's access conditions (planning, analysis requests, etc.). He/she undertakes not to reserve instruments in an inconsiderate manner and to bear in mind that this is a common tool which he/she cannot use without considering the other users. No cancellation of the

reservation of an instrument within 2 hours before the reservation will be authorized: the reservation will then be invoiced.

5.6 Services for third parties

Research units or organisations with a contract giving free access to the NMR facility's instruments undertake not to carry out analyses that are not part of their direct research activities. Analyses for a third party must not be invoiced in the name of an entity other than the NMR facility.

5.7 Valorization

Any internal or external user undertakes to acknowledge the NMR facility for the analyses performed on the instruments:

- a) The analyses were performed as a service only: recognition is expressed in the articles by the mention: " *NMR analyses were performed on the CPM NMR facility of Université de Lorraine* " in the " Materials & Methods "; and/or "*The authors would like to thank the CPM NMR facility of Université de Lorraine for its contribution to the present publication* " in the acknowledgements.
- b) The analyses involved specific developments and/or specific intellectual and/or technical participation by one or more of the NMR facility's staff, which had a significant impact on the progress of the work: the NMR facility's staff will be included in the list of co-authors. The position will be discussed with the authors.

Users are also required to inform the NMR facility manager and provide her with the references of publications including data obtained through the NMR facility.

Unless explicitly stated otherwise by the applicant, the service and the title of the project may be mentioned in the NMR facility's communication materials (activity reports, oral presentations, posters, brochures, etc.). The results will not be divulged by the NMR facility, except with the prior agreement of the applicant.

6. Acceptance of the charter

By signing this charter, the user undertakes to respect the conditions of access and work on the NMR facility, and more generally the various rules set out in this charter.

Any person who does not respect the commitments of this document, as well as the protocols in force on the NMR facility or any other recommendation concerning health and safety, may be denied access to the NMR facility by the manager.

Done at _____ , on _____ /_____ /_____

The person responsible for the NMR facility/equipment (name, signature)	The user (name, signature)

ANNEXE 1 – equipment list

Bruker Avance III 600 (Nancy)

Probe BBFO 5mm
Probe TBI 5mm
Probe BBO 10mm
Probe MAS DVT HXY 4mm
Probe MAS – LED HX 3.2mm
Probe MAS DVT HX 2.5mm
Probe MAS DVT HXY 1.3mm
Probe MicWB40 with coils 1H 25mm, 1H/13C 10mm, 1H/13C 5mm, 1H surface 2cm, 23Na 25mm
Probe MicWB57
Probe Diff30 with coils 1H 5mm, coil 1H/13C 5mm, coil 1H 10mm
Air cooler BCU-X
Rheology device RHEO-NMR

Bruker Avance III 400 « 400 direct » (Nancy)

Probe BBFO 5mm auto-tuning
Probe BBO 10mm : double resonance, nuclei 1H, 2H et X, direct detection of heteronuclei
Probe DUAL 10mm : double resonance, nuclei 1H, 2H et 19F
Probe 5mm : nuclei 1H and 2H
Air cooler BCU-05
SampleCase+ 60 position autosampler

Bruker Avance III 400 « 400 inverse » (Nancy)

Probe TBI 5mm
Probe BBI 5mm auto-tuning
SampleCase+ 60 position autosampler

Bruker Avance Neo Nanobay 400 (Metz)

Probe BBFO+ 5mm auto-tuning
SampleCase+ 60 position autosampler

Bruker Avance IIIHD 300 (Nancy)

Probe BBO 5mm
Probe BBI 10mm
Probe SEI High Temperature 5mm
Probe DUAL 5mm : double resonance, nuclei 1H, 2H and 13C
Probe DUAL 5mm : double resonance, nuclei 1H, 2H and 29Si
Probe MAS DVT HXY 4mm
Probe MAS VTN HX 2.5mm
Air cooler BCU-05

Bruker Avance Biospec 24/40 (Nancy)

Gradients BGA20s
BGA6s Gradient Insert
Volumetric Antennas Rapid Biomedical 1H 1.5cm 3.5cm, 5cm, 9cm, 16cm
Antenna « Litzcoil » Doty 15.5cm
Surface antennas 1H/13C and 1H/31P

ANNEXE 2 – schedules for the use of the "400 direct" and "400 inverse" at the Nancy site

planning d'utilisation du spectromètre 400MHz direct (sonde BBO)

(hors périodes de fermeture de la faculté des sciences)

	Lundi	Mardi	Mercredi	Jeudi	Vendredi	Samedi	Dimanche
8h -14h	Service commum	plein N_2 L2CM	L2CM	L2CM	Service commum	sur réservation	sur réservation
14h-18h	LERMAB	L2CM	L2CM	LERMAB	sur réservation	sur réservation	sur réservation
nuit	LERMAB	L2CM	L2CM	LERMAB	sur réservation	sur réservation	sur réservation

planning d'utilisation du spectromètre 400MHz inverse (sonde BBI)

(hors périodes de fermeture de la faculté des sciences)

accès libre: seulement expériences courtes en journée (8h-18h)

	Lundi	Mardi	Mercredi	Jeudi	Vendredi	Samedi	Dimanche
8h -14h	*accès libre*	*accès libre*	plein N_2 *accès libre*	*accès libre*	*accès libre*	sur réservation	sur réservation
14h-18h	*accès libre*	*accès libre*	*accès libre*	*accès libre*	Service commum	sur réservation	sur réservation
nuit	*accès libre (expériences longues)*	*accès libre (expériences longues)*	*accès libre (expériences longues)*	*accès libre (expériences longues)*	sur réservation	sur réservation	sur réservation

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1. The NMR NMR facility schedule is accessible via a web browser at this URL: https://resa-ijb.univ-lorraine.fr

2. To log in, use your UL ID (e.g. Dupont5) and password. If you are already registered as an authorized GRR user, go to the next step, otherwise you will be identified as a "visitor" and only consultation is possible. To make reservation requests and join the list of users, you must send a request with your "UL ID" to: cpm-plateforme-rmn-contact@univ-lorraine.fr;

3. The main screen displays a view of all spectrometers in the "NMR facility" domain in the form of a calendar (view of the current week)
The user can :
- choose the mode of display of the reservations (display "day" by clicking on the day, "week" by clicking on the week number, "month" by clicking on the month),
- select the day of the reservation in a calendar,
- choose the domain and the spectrometer of the domain to be displayed, here the domain is "NMR facility",
- By clicking on a reservation in the schedule, the user can view the details.

4. To reserve a spectrometer for a given time period, the user chooses the schedule ("day", "week" or "month" view) and the spectrometer to reserve. Then he clicks, in the schedule, on the small colored cross in the box that corresponds to the beginning of the reservation. A reservation form appears, the user must complete it before validating.
- Reservations are moderated and are made in the name of the connected person.
- It is important to fill in the "brief description" field with the name of your research unit (e.g. LEMTA, L2CM, LERMAB, ...) and your Name.
- As an option (without obligation), the "complete description" field can indicate the type of experiment: solid, liquid, nucleus (1H, 13C, 29Si, 27Al,...), etc ...
- Choose your laboratory in the list.
- Choose the start and end date of the reservation.
- The domain : here " NMR facility ".
- The spectrometer to reserve.
- The type of reservation: "Spectrometer reservation", the other parameters are reserved for the facility administrators.
- To validate the request, click on " Save ".

5. It is possible to modify or delete a reservation:
- As for the reservation, it is necessary to identify yourself to modify or delete a reservation.
- The persons authorized to delete or modify a reservation are: the owner of the reservation himself, on the condition that the reservation has not been passed, the manager of the resource, the general administrator of GRR or the restricted administrator of the concerned domain.
- You must click on the reservation you wish to modify or delete and then, in the new page that opens, click on the link corresponding to the desired action (delete or modify).

ANNEXE 3 – recommendations for sample preparation

The performance of high-resolution NMR experiments requires first of all a good quality sample that meets a number of constraints.

Selection of NMR tubes

NMR tubes must be of good quality, clean and dry before use. The sensitivity, particularly in proton NMR, is such that any impurities will be detected and may affect the quality or interpretation of your analysis. The minimum length of these tubes should be 178 mm. The university's central shop offers three qualities of tubes that are compatible with the equipment. High flow" tubes in boxes of 100 (MAG-VTRMN05 - Wilmad WG-1000-7) are sufficient for fully automated autosampler analyses. Standard tubes (MAG-VTRMN01 - Wilmad WG-1228) are suitable for most experiments and may give slightly better magnetic field homogeneity. The high quality tubes (MAG-VTRMN011 - Wilmad 527-PP) provide the best magnetic field homogeneity when very high resolution is desired.

Filling tubes

The resolution of an NMR spectrum depends on the setting of the homogeneity of the magnetic field in which the sample will be immersed. This adjustment is delicate and must be carried out for each sample, as it is strongly dependent on the height of the liquid in the tube. To optimize the automatic adjustment of the homogeneity, it is therefore recommended to use a constant filling height of 40mm (corresponding to approximately 600µL of solution for 5mm tubes).).

Choice of deuterated solvent

It is necessary to use deuterated solvents in NMR to stabilize and adjust the homogeneity of the magnetic field by observing the deuterium. Deuterated products are never at 100% isotopic enrichment. The non-deuterated solvent residue will lead to a proton signal which sometimes overlaps with the signals of the molecule or molecules of interest.

Solute concentration

NMR is not a very sensitive technique. It is therefore necessary to concentrate your samples. This statement is valid for measurements of nuclei with low isotopic concentrations such as carbon 13. A concentration of 0.6 $mol.l^{-1}$ allows rapid analysis (15 to 20 minutes) in ^{13}C. This concentration corresponds to 40 mg of a compound of molar mass 100 $g.mol^{-1}$ in 580 µl (volume corresponding to a 5 mm tube filled with 4 cm of liquid). If your concentration is much lower than this value, you will need to allow more time for the measurement.

ANNEXE 4 – non-exhaustive list of experiments that can be performed on the two self-service 400MHz spectrometers

- Standard ^{1}H experiment. (**PROTON**)

Approximate duration: 2 minutes

The default spectral window ranges from -4 to 16ppm.

- ^{1}H experiment with solvent removal. (**WATERSUP**)

Approximate time: 2 min

This experiment using gradients allows to obtain the spectrum of a solute in low concentration (~ 1mM) in light water whose signal must be suppressed.

- Experiment 2D COSY. (**COSY45SW**)

Approximate duration: 20 min

This is the simplest of the homonuclear correlation experiments which allows to obtain, via scalar coupling, the connectivity between protons.

- Experiments 1D and 2D NOESY and ROESY.

Approximate duration: depending on the system studied (usually one night)

The NOESY experiment allows the spatial proximity of protons to be highlighted. It can be a tool of choice to elucidate a structure.

- Standard ^{13}C experiment decoupled from the proton. (**13C CPD**)

Approximate duration: 20 minutes to several hours depending on the concentration of the solute

This is the basic experiment for the observation of ^{13}C. Quaternary carbons or carboxyls may be more difficult to measure due to slow relaxation combined with lower sensitivity. The spectra obtained are not quantitative. This experiment should be carried out as a priority on the "400 direct" which has a better sensitivity for the direct observation of ^{13}C.

- Experiment Attached Proton Test. (**13C APT**)

Approximate duration: from 40 minutes to several hours depending on the concentration of the solute

This experiment allows you to differentiate between carbons carrying 1 or 3 protons and quaternary carbons or carbons carrying 2 protons. The former will lead to a "negative" signal and the latter to a "positive" signal in the spectrum. This experiment should be carried out as a priority on the "400 direct", which is more sensitive for the direct observation of ^{13}C.

- Quantitative experiment ^{13}C decoupled from the proton.

Approximate duration: from 1 to several hours depending on the concentration of the solute

This experiment allows quantitative measurement of ^{13}C. It requires a much longer experimental time. A particular preparation of the sample (addition of a relaxing agent) may be necessary. This experiment should be carried out as a priority on the "400 direct", which is more sensitive for the direct observation of ^{13}C.

- Standard ^{19}F experiment. (**^{19}F**)

Approximate duration: 10 minutes

This is the basic experiment for observing ^{19}F.

- Standard ^{31}P experiment decoupled from the proton. (**^{31}P**)

Approximate duration: from 10 minutes to several hours depending on the concentration of the solute

This is the basic experiment for the observation of ^{31}P. As the probe of the "400 direct" spectrometer is optimized for the ^{31}P and ^{13}C nuclei in direct observation, it is preferable to carry out these experiments there.

- 2D Directly Bound ^{1}H-X Core Correlation Experiment (**HSQCETGP**)

Approximate duration: 1 hour

The HSQC experiment with the use of gradients allows to obtain in a short experimental time with low concentrations (~50 mM) the correlation map between chemically bound proton(s) and X-nucleus (via the 1J couplings between protons and X-nucleus). It is very useful to unambiguously identify the different resonances of the proton and X-ray spectra. This experiment should be carried out as a priority on the "400 inverse" which presents a better sensitivity for the ^{1}H.

- 2D correlation experiment ^{1}H-^{13}C not directly bound (**HMBCGP**)

Approximate duration: 3 hours

The HMBC experiment with the use of gradients allows to obtain in a short experimental time with low concentrations (~50 mM) the correlation map between proton(s) and non-chemically bound ^{13}C (via nJ couplings (n>1) between protons and carbon 13). This is a powerful tool for structure resolution and identification of quaternary carbons. This experiment should be carried out as a priority on the "400 inverse" which has a better sensitivity for ^{1}H observation.