

Automatic 400 MHz and 500 MHz NMR Regulations

(September 2024)

Revision of: February 2020

1. Introduction

The purpose of this document is to define the rules governing the use of Spectropole's 400 MHz and 500 MHz NMR instruments operating in automatic mode. These devices are programmed to deliver reliable results quickly, for the benefit of as many users as possible, and to make molecular chemistry research as fluid as possible. Their utilization statistics are impressive, with over 50,000 spectra recorded per year, and an occupancy rate (including maintenance) close to 350 days/year (24/24). To prevent any breakdowns and keep these precious tools in top condition, we ask everyone to adhere strictly to these rules. **Everyone must understand that any failure to comply with the rules or incivility will result in delays or even stoppages in the scheduling of experiments**, often leading to irritation and annoyance, increased maintenance costs, mobilization time for the service engineer and, in the case of the most serious breakdowns, service stoppage. Given the importance of our system, we therefore appeal to everyone's responsibility, especially that of our permanent researchers, to ensure that these basic rules, which have been collectively validated, are respected. **Each permanent researcher holding an account (or login) therefore undertakes to sign this document** and to have it read by any trainees, doctoral students or post-doctoral fellows who use their login and password after training.

2. General framework for use and operation of the automatic NMR facility

a. Training

All users, including account holders, must have completed a training course before using the spectrometers. Application to open an NMR account (permanent users) should be made to Mr Florian FERRER (florian.ferrer@univ-amu.fr).

Note: This rule will not apply to PhD students, post-docs, researchers and visiting academics who are already using equivalent spectrometers in their respective laboratories. Validation of their access to the service will be conditional on an assessment of their ability to use spectrometers correctly in automatic mode.

The training course is ONLY offered by Spectropole following a request via the following link: [Registration form for automatic NMR training - Fédération Sciences Chimiques Marseille](#). All permanent users and trainees must present themselves at the training session with the authorization form (available on the FSCM website) duly completed, and will then be listed and approved after validation of the course and signature of the form by the training engineer. Internships can also be carried out in English for foreign students. The protocol for using spectrometers running under IconNMR™ is available on the Spectropole website (spectropole.fr).

b. Tube quality

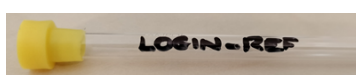
Tubes of poor quality, chipped or less than 20 cm long, or which do not fit the turbines supplied, will not be accepted. Please note that NMR tubes must not be dried in an oven, otherwise they will become brittle and deformed (risk of breakage and reduced spectral resolution).

Note: The majority of failures (> 80 %) observed on both spectrometers are due to non-compliance with this basic rule. Any failure to comply will result in sanctions (see sanctions chapter).

c. Samples

Samples must be prepared in the user's laboratory. No laboratory equipment will be allowed in the spectrometer room. In particular, **it is strictly forbidden to wear used laboratory gloves** (e.g. used during synthesis or other manipulations). The sample preparation protocol, available on the Spectropole website (spectropole.fr), must be followed. For specific needs, a bench in the preparation room is available on request in department 511. However, all traces of preparation must be removed and the bench must be left clean. To make it easier to identify your samples, please write your login and sample reference on the top of the tube using an indelible felt-tip pen (**Scheme 1**). Please also wipe your tubes clean before placing them on the autosampler, to avoid fingerprints which could compromise the analysis result.

Handle the turbines with care and **check the fit of the tubes**. No external elements, such as paper, should be introduced into the turbines to adjust a tube. In the event of a problem, please refer directly to your service manager.



Scheme 1. Tube marking illustration

3. How the spectrometers work

Spectrometers operate in 3 distinct modes: “day”, “night” and “weekend”. These modes are defined by well-defined time slots and programming rules, which are generally specific to the spectrometers in question (**Table 1**).

Table 1 : Time slots corresponding to the 3 programming modes (day, night and weekend) on the 2 spectrometers operating in automatic mode (400 MHz and 500 MHz)

Mode	Spectrometer 400 MHz	Spectrometer 500 MHz
« Day »	7:30 am – 7:00 pm	7:00 am – 7:00 pm
« Night »	7:00 pm – 7:30 am [D+1] (*)	7:00 pm – 7:00 am [D+1] (*)
« Weekend »	Friday 7:00 pm – Monday 7:30 am	Friday 7:00 pm – Monday 7:00 am

(*) These slots are subject to change depending on spectrometer occupation and possible breakdowns.

a. Mode « day »

The “day” mode is mainly dedicated to recording short-term experiments on sensitive nuclei (e.g. : ^1H , ^2H , ^{19}F , ^{31}P ...). On the 500 MHz, it is also possible to record experiments if there is a sufficient quantity of product ^{13}C and 2D (COSY and HSQC). On both spectrometers (400 MHz and 500 MHz), the order of programming on the respective sample changers prevails (position n , then position $n+1$, then position $n+2$...). In addition, to maintain a certain fluidity of operation for both spectrometers, and in particular to avoid excessive mobilization by a single login, the following programming rules have been put in place:

- * On the **400 MHz** spectrometer, only **8 minutes** of consecutive use are allowed **per login**
- * On the **500 MHz** spectrometer, only **15 minutes** of consecutive use are allowed **per login**

These maximum times apply to the programming by a user (*i.e.* by a login) of all experiments to be recorded consecutively on one tube, or on two tubes, or on three tubes. In each case, the user must add three empty positions on the feeder. These rules are listed in **Table 2** and illustrated in Appendix 2.

Note: As long as this rule is respected, there's nothing to prevent the same login from programming one (or more) other series of experiments, provided that i) the above rule is scrupulously respected, and ii) there are still positions available on the setter. The total programming time (over a day) per login is therefore not limited to 8 min on the 400 MHz or 15 min on the 500 MHz, but simply depends on the daily activity.

b. Mode « night »

In this mode, the progress of experiments depends on the time of sample deposit (first programmed is first served). For example: if you are the first to program yourself in night mode on position 8, even if another user programs a tube in night mode in position 2 after you, the sample in position 8 will go before the one in position 2. As a reminder, **the positions reserved for “night” mode are ONLY positions 1 to 20 for the 400 MHz spectrometer and positions 1 to 10 for the 500 MHz spectrometer**. To ensure that every user can benefit from this programming mode, which is particularly useful for recording long experiments (e.g. 2D NMR or 1D NMR on small quantities of products), the instructions given in **Table 2** must be followed. In addition, **it is forbidden to program experiments in both “day” and “night” modes for the same tube**. So, for example, if a user wishes to quickly check the quality of his product before launching a series of 2D experiments, he will first have to program a 1H spectrum in “day” mode, and then, if necessary, position the tube in “night” mode to record 2D experiments. Finally, **on an exceptional basis** (particularly during very busy periods), users may contact the NMR department engineer for authorization to program experiments in “night” mode on positions other than those normally reserved.

Note: In the event of failure of one of the two autosamplers, the “Night” mode of the other functional autosampler will be deactivated for the duration of the repair, in order to facilitate analysis.

c. Mode « weekend »

At weekends, spectrometers operate in “night” mode. The major difference is that, in this case, all the positions of the tuner are considered (instead of positions 1-20 for the 400 MHz or 1-10 for the 500 MHz). As a result, programming rules and instructions are similar to those for “night” mode (**Table 2**).

*Note: Failure to comply with these programming instructions will result in the **tubes concerned being deprogrammed** by Spectropole engineers.*

Table 2: Login programming rules for 400 MHz and 500 MHz NMR spectrometers.

Mode	400 MHz (AV400 Nano)	500 MHz (AV500)	Notes/Instructions
« Day »	Per login: 8 min. max for all experiments for 1, 2, or 3 tubes maximum programmed in sequence (See Appendix 2a)	Per login: 15 min. max for all experiments for 1, 2, ou 3 tubes maximum programmed in sequence (Voir Annexe 2b)	In all cases, leave 3 places free
« Night »	Per login: 3h for all experiments for 1 tube Up to 3 tubes programmed	Per login: 3h for all experiments for 1 tube Up to 2 tubes programmed	Remember: The « night » positions are numbered: - 1 à 20 (400 MHz) - 1 à 10 (500 MHz)
« Weekend »	Per login: 3h for all experiments for 1 tube	Per login: 3h for all experiments for 1 tube	-

d. Priority, sensitive products & kinetics

Users **must program and submit their samples in the order of the autosampler's progress**. In the event of rapid product deterioration, a priority request must be made by e-mail to the Spectropole engineer. **Priority is given during the day only**. It is punctual and does not exceed 48 hours. No priority is possible at night. Experiments on **labile products** or **kinetics** will be carried out by **appointment** and, subject to availability, on the 600 MHz spectrometer, at the same price as experiments on automatic spectrometers (contact the Spectropole NMR engineer by e-mail). If the spectrometer (600 MHz) is unavailable, or if the engineer is absent, these experiments can be carried out on automatic equipment by prior arrangement.

e. Tube recovery

Tubes removed from the autosampler must be placed in the boxes bearing the user's name. They must be retrieved promptly after analysis. Please note that tubes are emptied at the beginning of each month and **that tubes not recovered are destroyed within 1 month**.

f. Spectrometer parameters

Users cannot modify experiment parameters; only those visible in the IconNMR™ programming window can be changed (**Scheme 2**). Adaptation of a pulse sequence and/or access to other parameters can be requested from the engineer.



Scheme 2. Icon for accessing modifiable parameters in the IconNMR™ programming software.

g. Visualization of experiment progress

The status of the 400 MHz autosampler can be viewed by logging on to the Spectropole website, or by direct link with login and password to the following address: <http://172.16.51.134:8015/template-login.htm>

h. Data recovery

A server has been set up by Spectropole to enable users to retrieve spectra recorded on the various NMR spectrometers. The protocol for retrieving these data is available on the Spectropole website (spectropole.fr). For obvious security reasons, **it is strictly forbidden**

to retrieve data using a USB key or any other storage medium. Any user who fails to comply with this rule will be immediately and permanently excluded.







Please note that **the Spectropole server is not a data backup or storage server**. Users are strongly advised to retrieve and save their data as soon as it becomes available on the server. **Spectropole cannot be held responsible for the loss of data stored on its server.** When the server's maximum storage capacity is reached, the oldest data is deleted. Prior to this operation, a warning e-mail will be sent to all registered users. However, for reasons of confidentiality, data may be deleted at the user's request. In this case, please contact the Spectropole NMR department engineer. The server architecture has been designed to minimize the risk of hacking or data theft. Nevertheless, the Spectropole does not guarantee the security of its server. It cannot therefore be held responsible in the event of piracy, data theft or server disk failure.

i. [General informations](#)

Information on maintenance, rule changes, etc. is available on the home page of the Spectropole website and posted in the spectrometer room. In the event of important information, an e-mail will be sent to all permanent users of the Fédération Sciences Chimiques. The whiteboards available in the NMR rooms are only available to provide information to users. They are not intended to receive complaints, which should be forwarded by your team representative on the Spectropole user council. Finally, please note that the code for the entrance door to department 511 and the code for the NMR room door will be given to you once your course has been validated.

4. Safety

Before and as soon as you enter, please observe the posted safety signs and instructions. On an NMR spectrometer, an intense static magnetic field surrounds the magnet. This invisible field is the source of several hazards of which users must be aware. As the intensity of the magnetic field decreases sharply with distance from the magnet, ground markings have been placed around each magnet to delimit the zone from which the field begins to be intense. In this zone, under the effect of the magnetic field, any metal object becomes a projectile capable of seriously injuring a person or damaging equipment. Users entering the room where the equipment is located **must place all metal objects (keys, telephones, coins, etc.) in the basket provided for this purpose**. It is also strongly recommended to deposit any electronic or magnetic objects that could be damaged by the magnet's magnetic field. Intense magnetic fields also present a health hazard for users: **anyone with electromagnetic implants, a pacemaker or any other implanted electronic device, or anyone with hypersensitivity to magnetic and electromagnetic fields, is strictly forbidden access to the equipment (Scheme 3).**

	Strong magnetic field		High-frequency field Non-ionizing radiation
	Dangerous zone because of risk of damage to credit cards, magnetic ID cards, magnetic tapes, hard disks, etc.		Implants : Access forbidden to people with pacemakers, defibrillators, hearing aids, insulin pumps or other dosage devices
	Access forbidden to persons with metallic implants or any other metallic prosthesis in the body		No metal objects allowed in the area, such as: watches, coins, pens, cell phones, keys, computers, etc.

Scheme 3. Specific risks related to magnetic field

In the case of disability or particular illness, **a medical opinion from the AMU or CNRS preventive medicine department is required** to ensure that access to the equipment presents no danger to the user. In addition, any change in a user's state of health that no longer allows access to the NMR service must be reported to the engineers in charge. Before using the equipment, users are required to read the recommendations of the Institut National de Recherche et de Sécurité on exposure limits to static magnetic fields, available at: <http://www.inrs.fr/accueil/produits/mediatheque/doc/publications.html?refINRS=PR%2043>

On the other hand, spectrometer magnets contain large quantities of cryogenic liquids (liquid helium and nitrogen). These liquids are dangerous due to their extremely low temperature and the risk of asphyxiation in the event of sudden evaporation. A special notice prohibiting access to NMR rooms is displayed on days when liquid helium and nitrogen are being filled. Any user not complying with this notice will be excluded. Nitrogen and helium are non-toxic gases. However, in the event of a magnet quench, a large quantity of gas can be rapidly produced by evaporation. To avoid any risk of asphyxiation, oxygen levels in the NMR rooms are constantly monitored. **In the event of insufficient oxygen levels, a visual and audible alarm indicates that the department must be evacuated.** The evacuation must take place as quickly as possible, in a calm atmosphere, following the exits indicated by the signs and under the responsibility of the evacuation officer of department 511 - Spectropole (V. Monnier). In the event of an alarm outside working hours, **you must evacuate the department immediately and do not enter until further notice.**

Note: Trainees are under the full responsibility of their supervisors. In particular, supervisors must ensure that trainees and non-permanent staff for whom they are responsible do not access the automatic NMR service outside their working hours.

5. Operating anomalies, sanctions and complaints

a. Operating anomalies

Any anomaly must be reported immediately to the engineer and recorded in the event book available in the department.

b. Sanctions

Failure to comply with these rules will result in **financial compensation of €100 per day** the spectrometers are immobilized for repairs. This amount, subject to annual review, will be set by the users' council. In addition, if the malfunction triggered by the malicious user requires BRUKER to be called in for repair, an additional charge of **50% of the repair bill** will be made to the laboratory of the team responsible (for information: the average cost of a probe repair is around €5,000). Warnings will be sent by e-mail to the account holder, and the laboratory's financial contribution will be charged to the Spectropole invoice, which is issued every four months.

Furthermore, in the event of non-compliance with the programming rules decided collectively and described in the present rules, the user concerned will be sent an initial warning (personal e-mail with a copy to the team leader and unit director concerned) and, in the event of a repeat offence within 3 months, a flat-rate penalty of €100 per infraction will be charged directly to the invoice of the research team concerned.

c. Complaints

Any complaint relating to a disagreement with the department's engineers, contradictory interpretations of these rules, or challenges to the penalties imposed, should be addressed directly to the FSCM Director.

Dr Pierre THUREAU
Director
FSCM (UAR 1739)



APPENDIX 1: Account holder agreement (login)

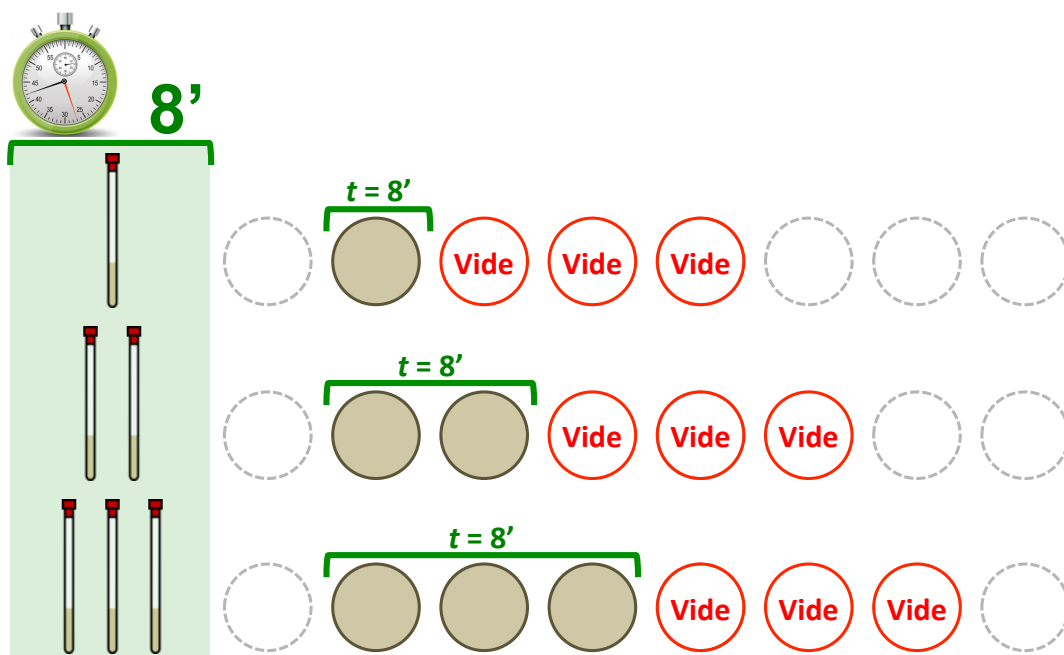
Last Name	:	
First Name	:	
UMR and Research Team	:	
Function	:	
Login	:	
Password	:	
Phone	:	
e-mail	:	

“I undertake to respect and ensure respect of these rules by trainees using my account (login). I also undertake to register any new trainee for a period of more than 4 months who is part of my team. I will inform Spectropole of any change in the composition of my team and the number of users with access to my account”.

Date and signature, preceded by the words “Read and approved”.

APPENDIX 2: Illustration of "day" mode programming rules

a) Spectrometer NMR 400 MHz



b) Spectrometer NMR 500 MHz

