Introductory Lecture

- NMR Facility Staff
- Magnet Hazards and Safety
- Sample Preparation
- Data Processing
- Facilities and Instrumentation
  - Open Access Facilities
  - NMR Submission Service
- On-line Resources: NMR web site
- Future training courses
1. NMR Staff

- **Facility Director:**
  - Dr Nick Rees

- **Service Manager:**
  - Dr James Montgomery

- **Service Technician:**
  - Caitlin Salter

- **NMR Officer:**
  - Coral Mycroft

nmrstaff@maillist.chem.ox.ac.uk
1. NMR Staff

- Facility Director:
  - Dr Nick Rees

- Service Manager:
  - Dr Coral Mycroft

- Service Technician:
  - Caitlin Salter

- NMR Officer:
  - Charlie Prentice

nmrstaff@maillist.chem.ox.ac.uk
Former NMR Staff

- Prof Tim Claridge
- Maria Marshall
2. Safety in the NMR laboratories

- Very Strong Magnetic Fields!
- Hazards to:
  - heart pacemakers
  - magnetic bank or ID cards
  - watches (non-LCD)
- Stray fields in corridors!
  - especially ground floor NMR
Safety Rules

- No laboratory coats in NMR labs.

- No metal objects to be taken into NMR labs.

- Sample breakages must be dealt with immediately
  - Inform the NMR staff if in any doubt
If you require any assistance or adjustments in relation to training and/or using this facility or if you have any concerns you would like to discuss beforehand, contact nick.rees@chem.ox.ac.uk
3. Sample Preparation

- Tubes and *deuterated* solvents from stores
- Tubes must be “Wilmad 507” or “Norell S400” grade (or equivalent) at least
- Tubes must not be scratched or broken
- Label tubes very carefully (Group & User Initials minimum)
- Solutions must be correct depth (4 cm)
- Solutions must be free from particulates
3. Sample Preparation

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- Tubes must be “Wilmad 507” or “Norell S400” grade (or equivalent) at least
- Tubes must not be scratched or broken
- Label tubes very carefully
- Solutions must be correct depth (4 cm)
- Solutions must be free from particulates
- Dry tubes carefully; acetone rinse then:
  - Leave on vacuum line for some hours
  - Lay flat in oven, 1 hour @ 100 °C max
NMR Tube Cap Colours for Organic Chemistry and Chemical Biology groups

<table>
<thead>
<tr>
<th>Group</th>
<th>Floor</th>
<th>Colour</th>
</tr>
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<tbody>
<tr>
<td>Professor E. A. Anderson</td>
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<tr>
<td>Professor J. Burton</td>
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<tr>
<td>Professor T. J. Donohoe</td>
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<td>PURPLE</td>
</tr>
<tr>
<td>Professor V. Gouverneur</td>
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<tr>
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<td>Professor M. G. Moloney</td>
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<td>Professor M. D. Smith</td>
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<td>Professor M. C. Willis</td>
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<td>Professor S. J. Conway</td>
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<tr>
<td>Professor S. G. Davies</td>
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<td>Professor I. McCulloch</td>
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<td>Professor J. Robertson</td>
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<td>Professor A. J. Russell</td>
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<td>lg</td>
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<td>Dr. M. J. Booth</td>
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<td>WHITE + BLACK</td>
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<td>Professor B. G. Davis</td>
<td>lg</td>
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<td>Professor C. J. Schofield</td>
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<td>ORANGE</td>
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<td>Professor T. Brown</td>
<td></td>
<td>BLUE</td>
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</tbody>
</table>

AQUA, BLACK, BLUE, FUCHSIA, GREEN, ORANGE, PINK, PURPLE, RED, SKY, WHITE, YELLOW
Sample masses required

- Rule of thumb for high-quality spectra (minimum):
  - 400 MHz Open-access spectrometers:
    - Proton & 2D COSY: 2 mgs
    - 2D H-C HSQC: 10 mgs
    - 1D Carbon: 20 mgs

- Please weigh your samples!!
How much is 10 mg?

NMR tube cap
Glycine Camphor CuSO₄
Data from all spectrometers can be downloaded *ONLY* from the on-line archive for off-line processing and local storage.

**NMR Store & archive:**
- Chemistry domain file sharing: `\\chem.ox.ac.uk\SRF\NMR`
- Macs: `smb://chem.ox.ac.uk/SRF/NMR`
Data Processing Software

- Windows and Macs:
  - **MestreNova**: 1D and 2D processing; platform independent
    - Chemistry site licence

- Windows and Macs:
  - **TOPSPIN**: Used on all spectrometers, 1D/2D NMR processing;
    - Free for academic use
Software installation

Departmental PCs and Laptops:

- **MestreNova**: Download latest version from Mestrelabs website. Copy and install licence from NMR server (\chem.ox.ac.uk\SRF\NMR\NMR Software\Mnova\)
  - [www.mestrelab.com](http://www.mestrelab.com)

- **Topspin**: Download from Bruker site and request licence:
  - [https://www.bruker.com/service/support-upgrades/software-downloads/nmr.html](https://www.bruker.com/service/support-upgrades/software-downloads/nmr.html)
Facilities operate at 4 levels:
- "open-access": automated instruments for all to use
- "hands-on": manual use of instruments for specifically trained users
- "submission service": analytical service provided by the NMR staff
- "research projects": collaborative projects involving the NMR staff/group
NMR in CRL
[Instrument nicknames shown]

Organic/Chem Biol Section

DPX200, AVF400
AVG400, AVH400
None
AVB400, AVB500, AVX500, NEO600, AV600, AV700

Inorganic Section

Venus400 + Mercury400
AVD500, Solids [HFX400 and HXY400]
Organic Chemistry and Chemical Biology
Instrumentation

10 research instruments @ 200-700 MHz
- 1 @ 200 MHz: Open Access $^1$H and $^{13}$C
- 3 @ 400 MHz: Open Access $^1$H, $^{13}$C, $^{19}$F, $^{31}$P & 2D
- 1 @ 400 MHz: Hands on multinuclear and VT work
- 2 @ 500 MHz: Hands on use & Service work
- 1 @ 600 MHz: Service work (NEO)
- 1 @ 600 MHz: Research projects
- 1 @ 700 MHz: Research projects
Automated 200 and three 400s
Provide rapid access to basic 1D & 2D $^1$H and $^{13}$C spectra, plus $^{19}$F and $^{31}$P
Available to all research workers
Spectra provided as PDF files and data on server

Training must be given by a member of the Analytical staff:
  ◦ Sessions will run Tuesday 3rd (pm), Thurs 5th (pm) and Fri 6th (am): Meet in CRL reception
  ◦ On-line booking and registration is required
Automated AVIII400 [Ground Floor]

Known as the AVG400

$^1$H, $^{13}$C, $^{19}$F, $^{31}$P
2D COSY
2D HSQC

file://chem.ox.ac.uk/SRF/NMR/AVG400/setup.html
Automated AVI400 [Ground Floor]

Known as the AVH400

$^1\text{H}, ^{13}\text{C}, ^{19}\text{F} \& ^{31}\text{P}$

2D COSY

2D HSQC

Faster for $^1\text{H}$ than AVF400 or AVG400

file://chem.ox.ac.uk/SRF/NMR/AVH400/setup.html
Automated AVIII 400 [1st Floor]

Known as the AVF400

$^1$H, $^{13}$C, $^{19}$F & $^{31}$P

2D COSY

2D HSQC

Generally very busy- only submit experiments you are sure are essential!

Check $^1$H only first

file://chem.ox.ac.uk/SRF/NMR/AVF400/setup.html
Semi-Automated DPX200 [1st Floor]

$^1$H NMR

Meant for fast $^1$H screening - first come, first served.

No robot operation.

file://chem.ox.ac.uk/SRF/NMR/DPX200/setup.html
High-field facilities

- Basement high-field NMR lab
- 400 and 500 MHz instruments available for specifically trained users ("hands-on" use). 600/700 MHz for bio-projects
- Training must be given by NMR staff
- Please enquire with NMR Staff if you wish to be trained
- On-line booking (intranet)- registration is required:
- [https://intranet.chem.ox.ac.uk/booking/default.html](https://intranet.chem.ox.ac.uk/booking/default.html)
NMR Submission Service

- Many routine 1D and 2D $^1$H, $^{13}$C, $^{19}$F, $^{31}$P, & $^{11}$B experiments can be performed using open-access 400 MHz

- Daily service provided by Dr Coral Mycroft and Caitlin Salter

- NMR Service uses 500 & 600 MHz instruments not 400

- Each sample must have electronic submission form (Word) and $^1$H spectrum (PDF) of same sample
NMR on-line Submission

Sample Number is unique for every NMR sample tube submitted and is used to track samples - every tube ID tag must be labelled with this number (at least)
The sample submission process will also be explained to you as part of your open-access training...
On-line resources

- NMR web pages:
  - http://nmrweb.chem.ox.ac.uk/
  - or through Intranet Link to Analytical Facilities on Chemistry homepage.
Future training courses

- Use of the Open-Access NMR Spectrometers & Service
  - Running this week - meet in CRL reception
  - Compulsory sessions - you must attend before using instruments or the NMR submission service.

- Mnova NMR Software Introductory Lecture
  - Single on-line lecture introducing main software features

- Modern NMR Spectroscopy for the Research Chemist
  - 8-lecture course providing overview of NMR techniques
  - This course can be found on the Oxford Canvas site at: https://canvas.ox.ac.uk/courses/54457

- CDT students
  - NMR training courses in Jan 2024: DO NOT SIGN UP FOR TRAINING THIS WEEK
Inorganic Chemistry
Instrumentation

- 5 research instruments @ 400-500 MHz
  - 1 @ 400 MHz: Open Access multinuclear
  - 1 @ 400 MHz: Open Access multinuclear
  - 1 @ 500 MHz: Hands on & Service multinuclear and VT work
  - 1 @ 400 MHz: Service Solid State HXY
  - 1 @ 400 MHz: Service Solid State HFX, microimaging & diffusion

- Access to 600 MHz: Service $^1$H and $^{13}$C
Automated AVIIIHD400 [2nd Floor]

Known as the Hg400

- 60 place autosampler
- $^1\text{H}, ^{19}\text{F}, ^{31}\text{P}$ to $^{13}\text{C}$
- $^1\text{H}–^1\text{H}, ^1\text{H}–^{13}\text{C}$ gradient selected 2-D experiments

Meant for fast daytime turnaround & longer overnight experiments

file://chem.ox.ac.uk/SRF/NMR/HG400/setup.html
Automated AVIII400 [2nd Floor]

Known as Venus400

- 60 place autosampler
- $^1$H, $^{19}$F, $^{31}$P to $^{13}$C
- $^1$H–$^1$H, $^1$H–$^{13}$C gradient selected 2-D experiments

Meant for fast daytime turnaround & longer overnight experiments

file://chem.ox.ac.uk/SRF/NMR/VENUS400/setup.html
Hands on AVIII500 [basement]

Known as the **AVD500**

- 24 place autosampler
- $^1\text{H}$, $^{19}\text{F}$ to $^{109}\text{Ag}$
- $^1\text{H} – ^1\text{H}$, $^1\text{H}$-$X$
- gradient selected 2-D experiments
- VT work

Booking required (on-line system)
Solid state NMR

- Service provided by Dr Nick Rees
- Stable samples provided as a solid (c.a. 200mg)
- Unstable samples can be packed in glove box
- Consult Nick Rees before submitting samples
- nick.rees@chem.ox.ac.uk
- Submit Samples via the Sample submission service
- Stable samples should be placed in the box through the basement NMR lab hatch
- For unstable samples provide email address on submission form.
Solid state NMR

Sample Submission Form: Word document

List experiments
And specify SSNMR

If needs to be packed in glove box give contact email address

The sample submission process will also be explained to you as part of your open-access training...
Known as the HXY400

- 4 & 1.9 mm Triple Magic Angle Spinning Probes ($^1$H, $^{19}$F, $^{31}$P to $^{15}$N)
- 4mm Low Gamma ($^{13}$C to $^{109}$Ag) Magic Angle Spinning Probe
- Wideline Deuterium Probe
- Goniometer probe for oriented samples
- Variable temperature capable (-150 to 150C)
Solid State AVIIIHD400WB [basement]

Known as the **HFX400**

- 3.2mm HFX Triple Magic Angle Spinning Probes ($^1\text{H}$, $^{19}\text{F}$, $^{31}\text{P}$ to $^{15}\text{N}$)
- 30mm Micro-imaging probe
- Diffusion probe
- Variable temperature capable (-150 to 150°C)
Inorganic Open-Access training:

- Use of the Open-Access NMR Spectrometers & Service
  - *Running this week* - *meet in CRL reception*
  - *Compulsory sessions* - you must attend before using instruments or the NMR submission service.

- Mnova NMR Software Introductory Lecture
  - Single on-line lecture introducing main software features

- CDT students
  - *NMR training courses in Jan 2024*: *DO NOT SIGN UP FOR TRAINING THIS WEEK*
Instrumentation Access

Organic Chemistry and Chemical Biology
- AVF400
- AVG400
- AVH400
- DPX200
- AV400

Undergraduate Teaching Lab.

Trained users “hands-on”
- AVB400
- AVB500
- AVX500
- NEO600
- AV600
- AV700

NMR Service & Research

Inorganic Chemistry
- Mercury 400
- Venus 400

All Chemists “open-access”

Trained users “hands-on”
- AVD500
- HXY400
- HFX400

Solid-state
NMR training for new researchers

1: Register as an Organic Section NMR user:
https://forms.office.com/e/sYyFxBKJ7e or
1: Register as an Inorganic Section NMR user:
https://forms.office.com/r/h30g6LxEsT

2: Sign up for a training session
Select Organic Section NMR: or Inorganic Section NMR:
https://outlook.office365.com/owa/calendar/SRFInductions2023@UniOxfordNexus.onmicrosoft.com/bookings/

Meet for training in CRL reception

To arrange training external users should email:
nmrstaff@maillist.chem.ox.ac.uk

All the above links can be found in these slides on-line at:
http://nmrweb.chem.ox.ac.uk/
QR for training sign-up sheets
OPEN ACCESS TRAINING: NMR SERVICE

Coral Mycroft
(soon to be) NMR Service Manager
NMR Facility – 4 Levels Of Analysis

Open Access
- Walk up use at any time of the day
- Common 1D/2D experiments: $^1\text{H}$, $^{13}\text{C}$, $^{19}\text{F}$, $^{31}\text{P}$, COSY, HSQC
- Fast turnover and hence rapid sample throughput
- Ground/first floors: Organic
- Second floor: Inorganic

NMR Service
- A submission service where researchers may submit samples for the NMR staff to run on the basement instruments.

Hands on
- Users can book the basement systems for manual/automated operation.

Research Projects
- Collaborative projects involving the NMR staff/group
A Typical Procedure Of Analysing A Sample Using NMR

Check the sample quality
- Open-access spectrometers to collect a basic 1D $^1$H NMR spectrum
- Check structure corresponds to what you expect, as well as the integrity and quality of the sample

Collect further data
- Open-access spectrometers to characterise the molecules: 1D and 2D spectra
- Always assess the $^1$H NMR spectrum first

Further experiments (if necessary)
- If the data collected is insufficient, you can consider submitting the sample to the NMR service / use the hands-on instruments (if trained)
Why Use The NMR Service?

Availability of experiments
- Other nuclei: $^2$H, $^7$Li, $^{27}$Al
- 2D experiments: HMBC, NOESY, ROESY
- More specialised experiments: DOSY, variable temperature

Dilute samples
- Walk-up instruments do not allow you to adjust any experiment parameters
- The NMR service will adjust experiment parameters so acceptable spectra is obtained.

Higher field spectrometers
- 500 – 700 MHz

Solid state samples
Six Steps To Use The NMR Service
for Solution-State NMR

(solid-state mentioned later)
Step 1: NMR Sample Preparation

Sample Tubes
- 5 mm high-quality tubes: Norell 400S, Wilmad 507-PP, or New Era MP5 at least
- Cracked, scratched or broken tubes will not be accepted

Solvents
- Use a deuterated solvent
- Solvent depth for 5mm tubes should be between 4.0 - 4.5 cm (500 - 600 μL)

Quantity of sample
- $^1$H / $^{19}$F: 1-10 mg for the 600 MHz
- $^{13}$C: At least 10+ mg for the 600 MHz (ca. 50+ mg should be run on the 400s)
- $^{31}$P: 10 mg

Labelling of NMR tube
- Use the correct NMR cap colour
- Label cap/tube with initials, solvent and group
Step 2: Acquire A $^1$H NMR And Convert Spectrum To A PDF.
Step 3: Generate Submission Number

- Unique number associated with a NMR sample
- Three distinct samples = three unique numbers

NMR Facility Website
https://nmrchem.web.ox.ac.uk/home
The NMR facility housed in the Chemistry Research Laboratory, University of Oxford is one of the largest available to research chemists in the UK.

It houses thirteen solution-state and two solid-state FT NMR instruments with proton operating frequencies ranging up to 700 MHz, which are capable of running most experiments of interest to the research chemist. The facility supports the full range of chemical sciences research across the department and university including synthetic organic and inorganic chemistry, supramolecular chemistry, chemical biology, enzymology, metabolomics, catalysis and materials science.

The NMR facility is managed by Dr Nick Reeve and is operated and maintained by four members of staff in total.

⚠️ Safety notice

There are specific safety hazards associated with the intense stray magnetic fields in the vicinity of the NMR instruments which may affect or interfere with:
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https://nmrchem.ouo.ox.ac.uk/submission-service
Submission service

We run a submission service where researchers, both inside and outside the Chemistry department, may submit samples for the NMR staff to run on the basement instruments.

In our quest towards a more sustainable lab we have designed new NMR service labels which can be found here.

Internal samples (i.e. researchers in Chemistry)
External samples (i.e. researchers from other departments)
Submission service

We run a submission service where researchers, both inside and outside the Chemistry department, may submit samples for the NMR staff to run on the basement instruments.

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Internal samples (i.e. researchers in Chemistry)

To submit a sample for service analysis, please:

1. Visit the sample submission webpage and fill in the form. This form will assign you a submission number — note this down.
2. Download and complete the NMR submission form [Word], using the submission number assigned previously. Please feel free to provide any extra details here if necessary, such as unusual chemical shifts.
3. Place the completed form in the "NMR Submissions" folder in the NMR data server; this can be accessed at `\chem.oxy.ac.uk\SRF\NMR\NMR Submissions` (please see here for instructions on how to access this folder if needed). Forms should be named in the following manner:
   `<submission number>_<name>_<supervisor initials>`.
   Note that paper forms will not be accepted!
4. In the same folder, place a PDF copy of the 1D 1H spectrum of the same sample you are submitting, using the following name:
   `<submission number>_<name>_<supervisor initials>_1H.pdf`
5. Bring the sample down and place it either on the sponge rack or inside the small fridge as per the location indicated on the submission form. Both of these can be accessed via the hatch to the side of the NMR lab entrance.
   If your sample is unstable or requires special attention, choose "Request" as the location and do not bring down the sample; a member of the NMR staff will contact you to arrange for this.

After a sample has been submitted, you will find the following:

1. Your submission form has been removed from the NMR Submissions folder. This is normal and indicates that it has been processed by a member of the NMR staff.
2. The completion status on the submission webpage is updated to "Yes", and the spectrometer(s) used to run the sample indicated next to it.
NMR Service Sample Submission System

Please complete this form to add a sample to the NMR service system. You MUST ALSO complete a paper submission form to accompany each sample.

Your name: __________________________ Your Group: __________________________

Project Code / Charge Account: __________________________

* see Finance if you are not sure what this is.

Your Sample NMR Experiments: __________________________ eg, (¹H, ¹³C, HSQC, APT, COSY, ¹⁹F, NOE, HMBC, DEPT, ROESY, TOCSY, NOESY, etc)

Submit

Here are the most recent submissions and their service status

<table>
<thead>
<tr>
<th>Date Submitted</th>
<th>Name</th>
<th>Research Group</th>
<th>Experiments Required</th>
<th>Sample Number</th>
<th>Completed</th>
<th>Instrument</th>
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<td>Maddie Hindson</td>
<td>MDS</td>
<td>¹H, ¹³C, COSY, HMBC, HSQC pls</td>
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<td>28/9/2023</td>
<td>Zhu Xin Zhang</td>
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<td>¹H, ¹³C, HMBC please</td>
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<td>Nicole Lewis</td>
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NMR Service Sample Submission System

Please complete this form to add a sample to the NMR service system. You MUST ALSO complete a paper submission form to accompany each sample.

Your name: Coral Mycroft
Your Group: OTHER
Project Code / Charge Account: DMT12345

* see Finance if you are not sure what this is.

Your Sample NMR Experiments: 1H, 13C, COSY, HSQC, APT, COSY, 19F, NOE, HMBC, DEPT, ROESY, TOCSY, NOESY, etc

Submit

Here are the most recent submissions and their service status

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NMR Service Sample Submission System

Please complete this form to add a sample to the NMR service system. You MUST ALSO complete a paper submission form to accompany each sample.

Your name: Coral Mycroft  
Your Group: OTHER

Project Code / Charge Account: DMT12345

* see finance if you are not sure what this is.

Your Sample NMR Experiments: \((1^H, \, 1^C, \, COSY, \, HSQC)\), etc. (\((1^H, \, 1^C, \, HSQC, \, APT, \, COSY, \, 19F, \, NOE, \, HMBC, \, DEPT, \, ROESY, \, TOCSY, \, NOESY, \, etc)\)

Submit

Here are the most recent submissions and their service status:

<table>
<thead>
<tr>
<th>Date Submitted</th>
<th>Name</th>
<th>Research Group</th>
<th>Experiments Required</th>
<th>Sample Number</th>
<th>Completed</th>
<th>Instrument</th>
</tr>
</thead>
<tbody>
<tr>
<td>28/9/2023</td>
<td>Maddie Hindson</td>
<td>MDS</td>
<td>((1^H, , 1^C, , COSY, , HSQC))pls</td>
<td>71800</td>
<td></td>
<td></td>
</tr>
<tr>
<td>28/9/2023</td>
<td>Maddie Hindson</td>
<td>MDS</td>
<td>((1^H, , 1^C, , COSY, , HSQC))pls</td>
<td>71799</td>
<td></td>
<td></td>
</tr>
<tr>
<td>28/9/2023</td>
<td>Maddie Hindson</td>
<td>MDS</td>
<td>((1^H, , 1^C, , COSY, , HSQC))pls</td>
<td>71798</td>
<td></td>
<td></td>
</tr>
<tr>
<td>28/9/2023</td>
<td>Zhihui Zhang</td>
<td>CIS</td>
<td>((1^H, , 15C, , HMBC)) please</td>
<td>71797</td>
<td></td>
<td></td>
</tr>
<tr>
<td>27/9/2023</td>
<td>Marta Serfini</td>
<td>SJC</td>
<td>((1^H, , 15C, , COSY, , HSQC)) please</td>
<td>71795</td>
<td></td>
<td></td>
</tr>
<tr>
<td>27/9/2023</td>
<td>Nicholas Lee</td>
<td>IP</td>
<td>((1^H, , 15C, , COSY, , HSQC, , HMBC))</td>
<td>71796</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
NMR Service Sample Submission System

Please complete this form to add a sample to the NMR service system. You MUST ALSO complete a paper submission form to accompany each sample.

Your name: 
Your Group: 
Project Code / Charge Account: 

* see finance if you are not sure what this is.

Your Sample NMR Experiments: eg, (¹H, ¹³C, HSQC, APT, COSY, ¹⁹F, NOE, HMBC, DEPT, ROESY, TOCSY, NOESY, etc)

Submit

Here are the most recent submissions and their service status

<table>
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<tr>
<th>Date Submitted</th>
<th>Name</th>
<th>Research Group</th>
<th>Experiments Required</th>
<th>Sample Number</th>
<th>Completed</th>
<th>Instrument</th>
</tr>
</thead>
<tbody>
<tr>
<td>28/9/2023</td>
<td>Coral Mycroft</td>
<td>OTHER</td>
<td>¹H, ¹³C, COSY, HSQC</td>
<td>71801</td>
<td></td>
<td></td>
</tr>
<tr>
<td>28/9/2023</td>
<td>Maddie Hindson</td>
<td>MDS</td>
<td>¹H, ¹³C, COSY, HMBC, HSQC, HSQC parasites</td>
<td>71800</td>
<td></td>
<td></td>
</tr>
<tr>
<td>28/9/2023</td>
<td>Maddie Hindson</td>
<td>MDS</td>
<td>¹H, ¹³C, COSY, HMBC, HSQC parasites</td>
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</tr>
<tr>
<td>28/9/2023</td>
<td>Zhuoxin Zhang</td>
<td>CI</td>
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<td>¹H, ¹³C, COSY, HSQC, HMBC please</td>
<td>71796</td>
<td></td>
<td></td>
</tr>
</tbody>
</table>
NMR Facility
Chemistry Department NMR Research Facility

Submission service
We run a submission service where researchers, both inside and outside the Chemistry department, may submit samples for the NMR staff to run on the basement instruments.

In our quest towards a more sustainable lab we have designed new NMR service labels which can be found here.

Internal samples (i.e. researchers in Chemistry)
To submit a sample for service analysis, please:

1. Visit the sample submission webpage and fill in the form. This form will assign you a submission number — note this down.

2. Download and complete the NMR submission form [Word], using the submission number assigned previously. Please feel free to provide any extra details here if necessary, such as unusual chemical shifts.

3. Place the completed form in the “NMR Submissions” folder in the NMR data server; this can be accessed at \chem.ox.ac.uk\SRF\NMR\NMR Submissions (please see here for instructions on how to access this folder if needed). Forms should be named in the following manner:
   <submission number>_<name>_<supervisor initials>.
   Note that paper forms will not be accepted!

4. In the same folder, place a PDF copy of the 1D 1H spectrum of the same sample you are submitting, using the following name:
   <submission number>_<name>_<supervisor initials>_1H.pdf

5. Bring the sample down and place it either on the sponge rack or inside the small fridge as per the location indicated on the submission form. Both of these can be accessed via the hatch to the side of the NMR lab entrance.
   If your sample is unstable or requires special attention, choose “Request” as the location and do not bring down the sample; a member of the NMR staff will contact you to arrange for this.

After a sample has been submitted, you will find the following:

1. Your submission form has been removed from the NMR Submissions folder. This is normal and indicates that it has been processed by a member of the NMR staff.

2. The completion status on the submission webpage is updated to “Yes”, and the spectrometer(s) used to run the sample indicated next to it.
Sample and experiment information (mostly)

Guidance on filling out the submission form

Personal information
**INTERNAL: NMR SERVICE SUBMISSION FORM FOR DEPARTMENT OF CHEMISTRY**

**CHEMISTRY RESEARCH LABORATORY**

**NMR SERVICE**

<table>
<thead>
<tr>
<th>Name</th>
<th>Status</th>
<th>CRL Lab:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coral Mycroft</td>
<td>D.Phil. Post Doc</td>
<td>800.120</td>
</tr>
</tbody>
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<table>
<thead>
<tr>
<th>Email:</th>
<th>Phone:</th>
</tr>
</thead>
<tbody>
<tr>
<td><a href="mailto:nmrstaff@mail1st.chem.ox.ac.uk">nmrstaff@mail1st.chem.ox.ac.uk</a></td>
<td>12345</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Group:</th>
<th>Supervisor’s Name:</th>
</tr>
</thead>
<tbody>
<tr>
<td>VG / CJS / HLA / DOH</td>
<td>D.Phil/Post Doc name</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>Submission Number:</th>
<th>Project Code/ Charge Account:</th>
<th>Submission Date:</th>
</tr>
</thead>
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<td>71801</td>
<td>DMT12345</td>
<td>28/09/2023</td>
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</tbody>
</table>

<table>
<thead>
<tr>
<th>Nuclei of interest:</th>
<th>Sample:</th>
<th>Solution Depths:</th>
</tr>
</thead>
<tbody>
<tr>
<td>1H, 13C</td>
<td>Rack</td>
<td>Max 4.5 cm</td>
</tr>
</tbody>
</table>

Structure

Experiments required (list ALL):

1H, 13C, COSY, HSQC

Nature of problem:

Standard structure assignment of new compound. Sample too dilute for 13C and HSQC on open access spectrometers.

<table>
<thead>
<tr>
<th>Mass supplied:</th>
<th>Solvent:</th>
</tr>
</thead>
<tbody>
<tr>
<td>&lt; 1 mg</td>
<td>CDCl3</td>
</tr>
</tbody>
</table>
Step 5: Place Completed Form And PDF In The NMR Submissions Folder In The NMR Data Server

Word: <submission number>_<name>_<supervisor initials>
71801_Mycroft XXX

PDF: <submission number>_<name>_<supervisor initials>_1H.pdf
71801_Mycroft XXX_1H

Solid-state samples: Place word document in the ‘Solid State’ folder. No PDF required.
Submission service

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In our quest towards a more sustainable lab we have designed new NMR service labels which can be found [here](#).

### Internal samples (i.e. researchers in Chemistry)

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3. Place the completed form in the "NMR Submissions" folder in the NMR data server; this can be accessed at `\chemox.ac.uk\SRF\NMR\NMR Submissions` (please see [here](#) for instructions on how to access this folder if needed). Forms should be named in the following manner:
   
   `<submission number>_name_supervisor initials>.`  

   Note that paper forms will not be accepted!
4. In the same folder, place a PDF copy of the 1D 1H spectrum of the same sample you are submitting, using the following name:

   `<submission number>_name_supervisor initials>_1H.pdf`

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After a sample has been submitted, you will find the following:

1. Your submission form has been removed from the NMR Submissions folder. This is normal and indicates that it has been processed by a member of the NMR staff.
2. The completion status on the [submission webpage](#) is updated to "Yes", and the spectrometer(s) used to run the sample indicated next to it.
Archiving NMR data

Spectra acquired on all NMR spectrometers are initially stored on the hard disks of the individual computers attached to the spectrometers, and cannot be accessed directly this way.

To obtain your NMR data, the spectra must be transferred to a central server (sometimes called the 'archive') which you can then access.

- **Open-access**: this is automatically done
- **Submission service**: this is automatically done, please email nmrstaff@mailist.chem.ox.ac.uk if you cannot find your data
- **Hands-on**: this is automatically done if you ran your experiments under IconNMR automation. If you ran them manually, then you must use the archive TopSpin command on each individual dataset in order to transfer them to the server.

Accessing the NMR server

On university computers (including group computers) — Windows

1. Log into Windows as any user in the CHEM domain. This can be done with either your own account (usually firstname.lastname) or a group account (usually xyzgroup).
2. Open any folder, click on the address bar (see screenshot below) and enter: `\chem.ox.ac.uk\SRF\NMR`

You will then see a list of spectrometer data folders, from which you can obtain your spectra.

Includes how to access on university and own computers (Windows/macOS)
Submission service

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In our quest towards a more sustainable lab we have designed new NMR service labels which can be found here.

+ Expand All

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   `<submission number>_<name>_<supervisor initials>`.

   Note that paper forms will not be accepted!

4. In the same folder, place a PDF copy of the 1D $^1$H spectrum of the same sample you are submitting, using the following name:

   `<submission number>_<name>_<supervisor initials>_1H.pdf`

5. Bring the sample down and place it either on the sponge rack or inside the small fridge as per the location indicated on the submission form. Both of these can be accessed via the hatch to the side of the NMR lab entrance.

   If your sample is unstable or requires special attention, choose "Request" as the location and do not bring down the sample; a member of the NMR staff will contact you to arrange for this.

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2. The completion status on the [submission webpage](#) is updated to "Yes", and the spectrometer(s) used to run the sample indicated next to it.
Step 6: Bring The NMR Sample Down To The Basement With A Completed Label
Step 6: Bring The NMR Sample Down To The Basement With A Completed Label

<table>
<thead>
<tr>
<th>SAMPLE NUMBER:</th>
<th>HOLDER:</th>
</tr>
</thead>
<tbody>
<tr>
<td>71801</td>
<td>FOR NMR SERVICE ONLY – LEAVE BLANK</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>NAME:</th>
</tr>
</thead>
<tbody>
<tr>
<td>Coral Mycroft</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>GROUP:</th>
<th>DATE:</th>
</tr>
</thead>
<tbody>
<tr>
<td>HLA/ HB/ SJC</td>
<td>28/09/2023</td>
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</table>

<table>
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<tr>
<th>COST CODE:</th>
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<tbody>
<tr>
<td>DMT12345</td>
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<table>
<thead>
<tr>
<th>SOLVENT:</th>
<th>STORAGE (RACK / FRIDGE):</th>
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<tbody>
<tr>
<td>CDCl₃</td>
<td>Rack</td>
</tr>
</tbody>
</table>

<table>
<thead>
<tr>
<th>EXPERIMENTS:</th>
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</thead>
<tbody>
<tr>
<td>¹H, ¹³C, COSY, HSQC</td>
</tr>
</tbody>
</table>
Six Steps To Use The NMR Service for Solid-State NMR

1. Consult Nick Rees before submitting samples
   - Nick.rees@chem.ox.ac.uk

2. Prepare the NMR sample
   - Stable samples provided as a solid (c.a. 200 mg)
   - Unstable samples can be packed in a glove box

3. Generate a unique submission Number via the NMR Facility Website

4. Complete submission form

5. Place submission form in the Solid-State submission folder on the Q drive

6. Stable samples should be placed in the box through the basement NMR lab hatch

https://nmrchem.web.ox.ac.uk/
Next Steps: NMR Facility Staff Take Over

The NMR facility will check the sample, paperwork and PDF of the submitted sample
- Everything is correct: schedule the analysis
- Issue with sample/paperwork/PDF: analysis will not be scheduled

When analysis is being run, the submission form and PDF will be removed from the NMR Submissions folder

Turnover is dependent on the current demand of the NMR Service and the analysis requested

Once complete, the status on the submission webpage is updated to “Yes”, with details of which spectrometer was used to run the sample

<table>
<thead>
<tr>
<th>Date Submitted</th>
<th>Name</th>
<th>Research Group</th>
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<th>Sample Number</th>
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<th>Instrument</th>
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</thead>
<tbody>
<tr>
<td>28/9/2023</td>
<td>Coral Mycroft</td>
<td>OTHER</td>
<td>1H, 13C, COSY, HSQC</td>
<td>71801</td>
<td>Yes</td>
<td>AVX500</td>
</tr>
</tbody>
</table>
Next Steps: NMR Facility Staff Take Over

At this point you can obtain the data from the NMR data server. This data is organised by instrument and then by your research group initials. Each individual dataset is then named according to the following:

<initials> <five-digit submission number> <date in DDMM format>

cm718012909

The sample will be placed in the returns rack/fridge (solution) or completed tub (solid)
Why Hasn’t My Sample Been Run?

Check the submission system

If no information, the sample has yet to be run
- Service is very busy
- Analysis requires a specific spectrometer

If system says aborted, either an element of the paperwork or sample is incorrect
- Correct the error and re-submit the sample/paperwork
- Depending on how busy the service is, we may email you stating what is incorrect for you to fix

If it’s been >1 week and you’ve requested only conventional 1D and 2D experiments, you can email us: nmrstaff@maillist.chem.ox.ac.uk
NMR Facility Members

nmrstaff@maillist.chem.ox.ac.uk

Nick
Head of NMR

Caitlin
NMR Research Technician

Coral
NMR Service Manager

Charlie
NMR Officer
Training And Other

Providing you have registered, you are now trained to the open access NMR spectrometers on the relevant floors

- Ground/first floors: Organic
- Second floor: Inorganic

Use of the basement spectrometers in ‘hands on’ mode?

- Requires a separate 60-90 minute training
- Part 2 users are not allowed, unless their project has a significant NMR element

Always ask for help if you are unsure!
Talk to us in person, or contact the NMR staff mail list for all enquiries.

nmrstaff@maillist.chem.ox.ac.uk