

NMR@CHEM.OX

Chemistry Research Laboratory

# NMR Spectroscopy Facility Introductory Lecture

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&

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<https://nmr.chem.ox.ac.uk/>

# Introductory Lecture

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

# 1. NMR Staff (2023)

- ▶ Facility Director:
  - Dr Nick Rees
- ▶ Service Manager:
  - Dr Coral Mycroft
- ▶ Research Technician:
  - Caitlin Salter
- ▶ NMR Officer:
  - Charlie Prentice



Nick



Caitlin



Coral



Charlie

[nmrstaff@maillist.chem.ox.ac.uk](mailto:nmrstaff@maillist.chem.ox.ac.uk)

# 1. NMR Staff (2024)

- ▶ Head of Inorganic and Solid State NMR:
  - Dr Nick Rees
- ▶ Head of Organic and Biological NMR:
  - Dr Harry Mackenzie
- ▶ Service Manager:
  - Dr Coral Mycroft
- ▶ Research Technician:
  - Caitlin Salter

[nmrstaff@maillist.chem.ox.ac.uk](mailto:nmrstaff@maillist.chem.ox.ac.uk)



Nick



Harry



Coral



Caitlin

# Former NMR Staff



- Prof Tim Claridge



- Maria Marshall



- James Montgomery



- Charlotte Prentice

## 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

# Accessibility in the NMR laboratories

- ▶ 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

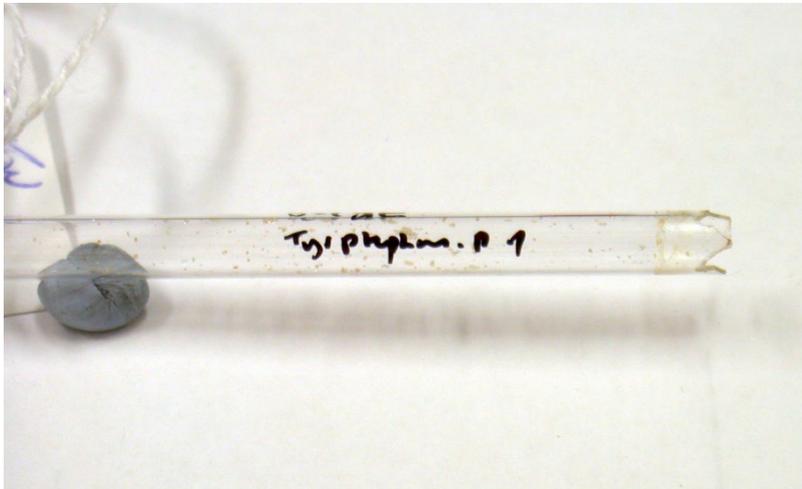
[nick.rees@chem.ox.ac.uk](mailto:nick.rees@chem.ox.ac.uk)

or

[harry.mackenzie@chem.ox.ac.uk](mailto:harry.mackenzie@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 (**see future slide**)
  - ▶ Solutions must be correct depth (4 – 4.5 cm)
  - ▶ Solutions must be free from particulates
- 



# 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
- ▶ 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

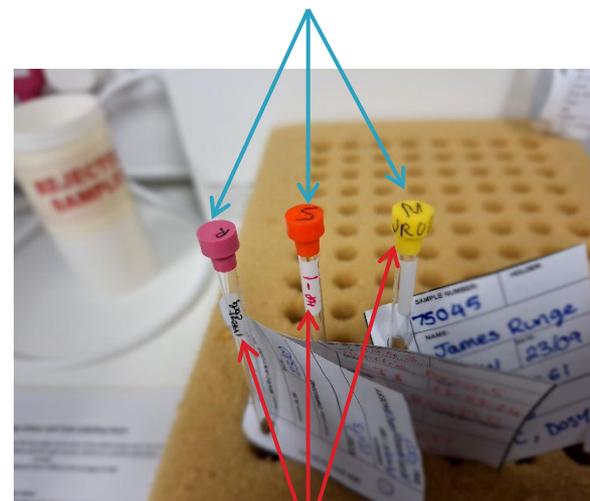
# NMR Cap Colour and Tube Labelling Rules

- ▶ The following rules are in place for the safety of all users of the NMR facility and must be followed. This includes NMR tubes used for open-access, hands-on, and the submission service.
  - ▶ Tube caps may be ordered from VWR on R12 as bags of 100.
- ▶ Rules:
  - All groups must use their group cap colour
  - The top of the cap must be clearly labelled with the surname initial of your group's academic
  - Your own name/initials must be clearly labelled on the tube
  - The solvent used must be clearly labelled on the tube
  - If you are using a J Young's tube, you may use any colour cap but the full group initials must be written on, as well as the other above information

# NMR Tube Cap Colours for Organic Chemistry and Chemical Biology groups

Research Group	Colour	Initial
E. A. Anderson	White	A
H. L. Anderson	Sky	A
H. Bayley	Pink	B
T. Brown	Blue	B
J. W. Burton	Yellow	B
S. J. Conway	Aqua	C
D. J. Dixon	Black	D
T. J. Donohoe	Purple	D
S. P. Fletcher	Sky	F
V. Gouverneur	Pink	G
I. McCulloch	Red	M
F. Probert	Purple	P
Y. Qing	Yellow	Q
P. Rabe	Red	R
J. Robertson	Orange	R
A. J. Russell	Fuchsia	R
C. J. Schofield	Orange	S
M. D. Smith	Blue	S
M. C. Willis	Aqua	W

Cap colour & Supervisor Initial

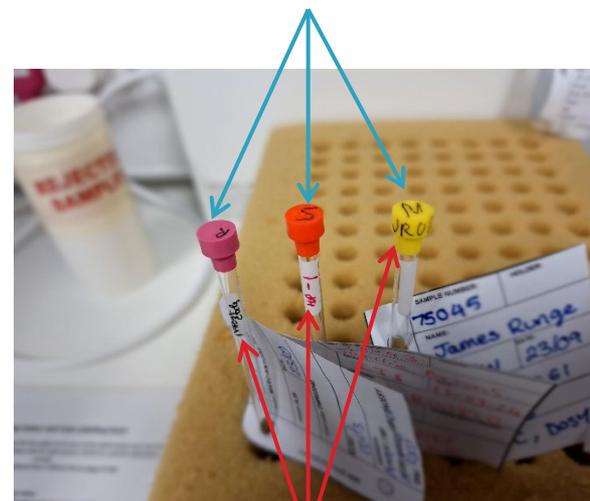


User Initials

# NMR Tube Cap Colours for Inorganic Chemistry groups

Inorganic Group	Colour	Initial
S. Aldridge	Red	A
P. D. Beer	Sky	B
J. J. Davis	Blue	D
S. Faulkner	Red	F
J. M. Goicoechea	Aqua	G
G. L. Gregory	Fuchsia	G
M. J. Langton	White	L
M. Mehta	Orange	M
M. Neidig	Pink	N
D. O'Hare	Green	O
E. Tsang	Blue	T
K. Vincent	Purple	V
C. K. Williams	Yellow	W
L. L. Wong	Green	W

Cap colour & Supervisor Initial



User Initials

# 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!!

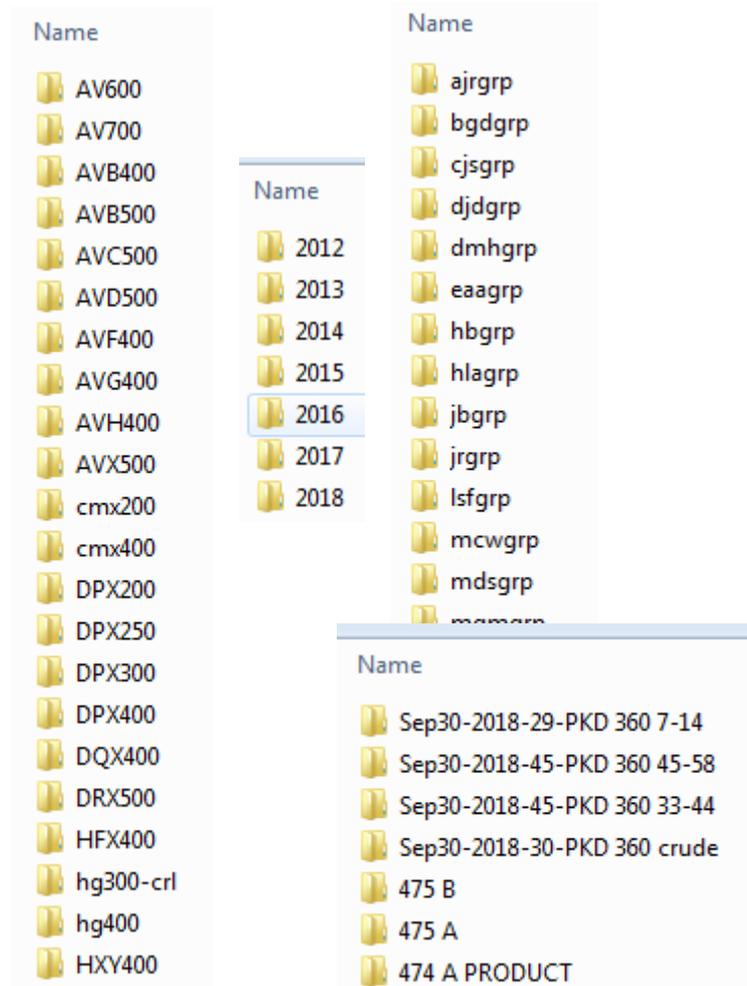
# How much is 10 mg?

NMR tube cap  
Glycine Camphor  $\text{CuSO}_4$



# 4. Data Processing & Storage

- ▶ 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>

# 5. How to use the facilities

- ▶ 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

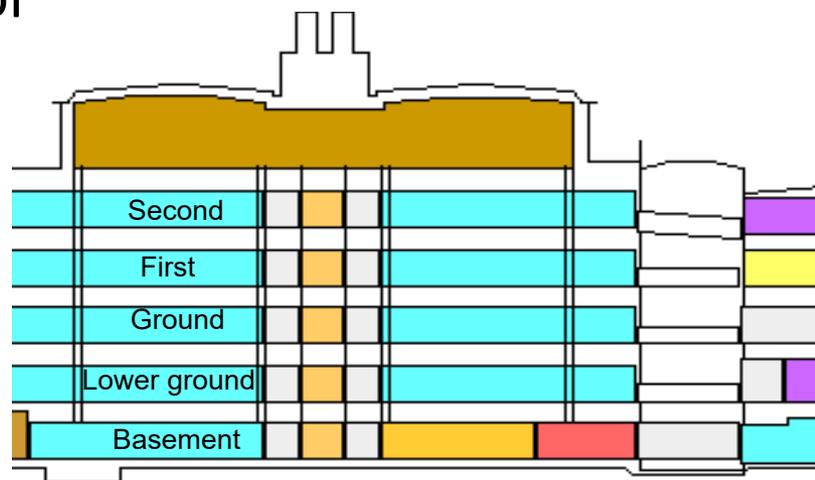
[Instrument nicknames shown]

## 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  $^1\text{H}$  and  $^{13}\text{C}$
  - 3 @ 400 MHz: Open Access  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{19}\text{F}$ ,  $^{31}\text{P}$  & 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 & Service work
  - 1 @ 700 MHz: Research projects & Service work

# Open access 200/400 MHz facilities

- ▶ Automated 200 and three 400s
- ▶ Provide rapid access to basic 1D & 2D  $^1\text{H}$  and  $^{13}\text{C}$  spectra, plus  $^{19}\text{F}$  and  $^{31}\text{P}$
- ▶ 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 Wednesday 2<sup>nd</sup> (pm), Thurs 3<sup>rd</sup> (pm) and Fri 4<sup>th</sup> (am): **Meet in CRL reception**
  - **Online booking and registration is required**

# Automated AVIII400 [Ground Floor]



Known as the AVG400

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

2D COSY

2D HSQC

<file:///chem.ox.ac.uk/SRF/NMR/AVG400/setup.html>

# Automated AVIII400 [Ground Floor]



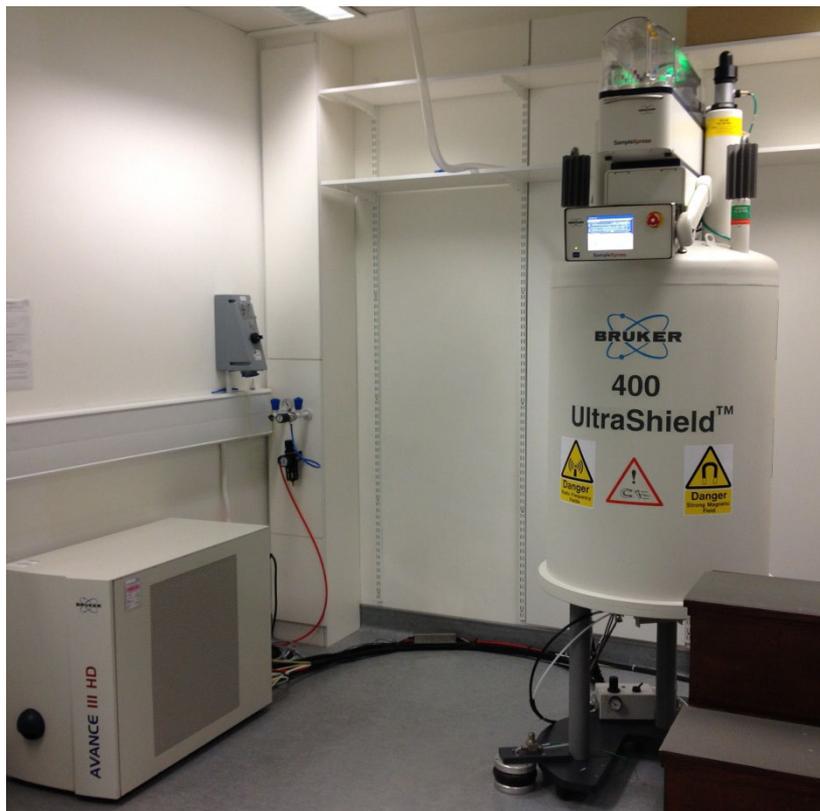
Known as the AVH400

$^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{19}\text{F}$  &  $^{31}\text{P}$   
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 [1<sup>st</sup> Floor]



Known as the AVF400

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

2D COSY

2D HSQC

Generally very busy- only submit experiments you are sure are essential!  
Check  $^1\text{H}$  only first

<file:///chem.ox.ac.uk/SRF/NMR/AVF400/setup.html>

# Semi-Automated DPX200 [1<sup>st</sup> Floor]



$^1\text{H}$  NMR

Meant for fast  $^1\text{H}$  screening-  
first come, first served.

No robot operation.

<file://chem.ox.ac.uk/SRF/NMR/DPX200/setup.html>

# Instrument status web pages

Instrument Booking | x | https://wessington... | x | Magnetic Resonance | x | Microsoft Forms | x | IconNMR History | x | Self-induced recoupling | x | Frontiers | Nuclear | x | IconNMR History | x | The Analytical Wa... | x | IconNMR History | x | +

File | Qz/AVG400/setup.html

Apps | BBC - Homepage | Tims | Department of Che... | Instrument Booking | NMR Facility | Sample Submission | Sample Managem... | Instruments | Journals | SMASH | NMR sites | Other sites | Bruker NMR | OxFile | Wikipedia | Reading list



IconNMR Automation Run Status:  
Day Experiments: 00:05  
Busy Until: Thu 12:07  
Night Experiments: 00:42

### Setup List

Holder	Status	Name	ExpNo	EXPERIMENT	User	Time	Title
1	Completed	Sep30-2021-1-DH_03_155_hexyl-dry	1	N h1acq.cr1 1H	mjbgrp	00:01:32	Instrument AVG400 Chemist DH Group MJB Project Account Code DMT00010
2	Completed	NB008	2	N h1acq.cr1 1H	mjbgrp	00:01:32	Instrument AVG400 Chemist NB Group MJB Project Account Code DQR00150 NB008 recrystallised
3	Completed	BF310-14-19	1	N h1acq.cr1 1H	djgrp	00:01:32	Instrument AVG400 Chemist BF Group DJD Project Account Code other
3	Completed	BF310-14-19	2	N p31dec.cr1 Phosphorus with 1H decoupling +200 to -200 ppm	djgrp	00:01:01	Instrument AVG400 Chemist BF Group DJD Project Account Code other
4	Completed	Sep30-2021-4-MLJM-B16	1	N h1acq.cr1 1H	sjcgrp	00:01:32	Instrument AVG400 Chemist MLJM Group SJC Project Account Code Other MLJM B16
6	Completed	Sep30-2021-6-TT-1.3	1	N h1acq.cr1 1H	hlagrp	00:01:32	Instrument AVG400 Chemist TT Group HLA Project Account Code Other
7	Completed	Sep30-2021-7-OJSC84_(1)_2	1	N h1acq.cr1 1H	sjcgrp	00:01:32	Instrument AVG400 Chemist OJS Group SJC Project Account Code BST00210
8	Completed	Sep30-2021-8-RT-9-867-pure	1	N h1acq.cr1 1H	djgrp	00:01:32	Instrument AVG400 Chemist Tanya Group DJD Project Account Code other
8	Completed	Sep30-2021-8-RT-9-867-pure	2	n c13acq_512.cr1 Carbon 512 scans	djgrp	00:14:33	Instrument AVG400 Chemist Tanya Group DJD Project Account Code other
9	Completed	Sep30-2021-9-SM-5-93_crd	1	N h1acq.cr1 1H	spfgrp	00:01:32	Instrument AVG400 Chemist SM Group SPF Project Account Code DMR00790 SM-5-93_crd
10	Completed	Sep30-2021-10-SM-5-94_crd	1	N h1acq.cr1 1H	spfgrp	00:01:32	Instrument AVG400 Chemist SM Group SPF Project Account Code DMR00790 SM-5-94_crd
11	Completed	Sep30-2021-11-MLJM-B16-Trit	1	N h1acq.cr1 1H	sjcgrp	00:01:32	Instrument AVG400 Chemist MLJM Group SJC Project Account Code Other MLJM B16 trit
12	Completed	Sep30-2021-12-RT-3-2	1	N h1acq.cr1 1H	cjsgrp	00:01:32	Instrument AVG400 Chemist RT Group CJS Project Account Code PT2
13	Completed	mm287	1	N h1acq.cr1 1H	hlagrp	00:01:32	Instrument AVG400 Chemist mm Group HLA Project Account Code dmr01490 Suzuki, Ac(OEt)2, PPh3, tol, 110 deg
14	Completed	Sep30-2021-14-ak1-690-3-crude1	1	N h1acq.cr1 1H	ajrgrp	00:01:32	Instrument AVG400 Chemist ak Group AJR Project Account Code DM8000
16	Completed	Sep30-2021-16-3332novo	1	N h1acq.cr1 1H	ajrgrp	00:01:32	Instrument AVG400 Chemist cjrj Group AJR Project Account Code DM8000 cjrj3332-novo
16	Completed	Sep30-2021-16-3332novo	2	N f19acq.cr1 Fluorine +100 to -250 ppm (16 scans)	ajrgrp	00:01:00	Instrument AVG400 Chemist cjrj Group AJR Project Account Code DM8000 cjrj3332-novo
16	Submitted	Sep30-2021-16-3332novo	3	n c13acq_512.cr1 Carbon 512 scans	ajrgrp	00:14:33	Instrument AVG400 Chemist cjrj Group AJR Project Account Code DM8000 cjrj3332-novo
16	Submitted	Sep30-2021-16-3332novo	4	n DEPT135.cr1 AV400 13C DEPT135	ajrgrp	00:03:41	Instrument AVG400 Chemist cjrj Group AJR Project Account Code DM8000 cjrj3332-novo
17	Completed	Sep30-2021-17-CA-2-071	1	N h1acq.cr1 1H	imgrp	00:01:32	Instrument AVG400 Chemist C Aitchison Group IM Project Account Code DMR01750
18	Completed	Sep30-2021-18-CA-2-073	1	N h1acq.cr1 1H	imgrp	00:01:32	Instrument AVG400 Chemist C Aitchison Group IM Project Account Code DMR01750
19	Completed	FM 07-61 AC	1	N h1acq.cr1 1H	spfgrp	00:01:32	Instrument AVG400 Chemist FM Group SPF Project Account Code DMR00790
20	Completed	Sep30-2021-20-ALR01	1	N h1acq.cr1 1H	ajrgrp	00:01:32	Instrument AVG400 Chemist Adam Pinto Group AJR Project Account Code P2 ALR01
21	Error	Sep29-2021-21-SL02-84-c	4	n HSQC.cr1	jrgp	00:05:05	Instrument AVG400 Chemist SL Group JR Project Account Code dm7900 fr 2
22	Completed	IM-001	1	N h1acq.cr1 1H	djgrp	00:01:32	Instrument AVG400 Chemist Iain McLauchlan Group DJD Project Account Code other
22	Submitted	IM-001	2	n c13acq_512.cr1 Carbon 512 scans	djgrp	00:14:33	Instrument AVG400 Chemist Iain McLauchlan Group DJD Project Account Code other
22	Completed	IM-001	3	N f19dec.cr1 Fluorine with 1H decoupling +100 to -250 ppm (16 scans)	djgrp	00:01:00	Instrument AVG400 Chemist Iain McLauchlan Group DJD Project Account Code other
22	Submitted	IM-001	4	n COSY.cr1	djgrp	00:05:05	Instrument AVG400 Chemist Iain McLauchlan Group DJD Project Account Code other
22	Submitted	IM-001	5	n HSQC.cr1	djgrp	00:05:05	Instrument AVG400 Chemist Iain McLauchlan Group DJD Project Account Code other
23	Completed	Sep30-2021-23-468x2-7-9	1	N p31dec.cr1 Phosphorus with 1H decoupling +200 to -200 ppm	bvlgp	00:01:01	Instrument AVG400 Chemist mls Group BVLP
24	Completed	Sep30-2021-24-468x2-10-12	1	N p31dec.cr1 Phosphorus with 1H decoupling +200 to -200 ppm	bvlgp	00:01:01	Instrument AVG400 Chemist mls Group BVLP
25	Completed	Sep30-2021-25-468x2-13-15	1	N h1acq.cr1 1H	bvlgp	00:01:32	Instrument AVG400 Chemist mls Group BVLP
25	Running	Sep30-2021-25-468x2-13-15	2	N p31dec.cr1 Phosphorus with 1H decoupling +200 to -200 ppm	bvlgp	00:01:01	Instrument AVG400 Chemist mls Group BVLP
26	Submitted	DSCH_04_019_col2_3_f22-28	1	N h1acq.cr1 1H	sfgrp	00:01:32	Instrument AVG400 Chemist DSneddon Group SF Project Account Code other 1H
27	Available	Sep30-2021-27-jr077_75_MIXED_SEC_FRAC	1	N h1acq.cr1 1H	hlagrp		Instrument AVG400 Group HLA
29	Submitted	Sep30-2021-29-468x2-16-18	1	N h1acq.cr1 1H	bvlgp	00:01:32	Instrument AVG400 Chemist mls Group BVLP
29	Submitted	Sep30-2021-29-468x2-16-18	2	N p31dec.cr1 Phosphorus with 1H decoupling +200 to -200 ppm	bvlgp	00:01:01	Instrument AVG400 Chemist mls Group BVLP
39	Error	Sep29-2021-38-2066SM meod	1	N h1sup.cr1 1H presat	corgrp	00:01:32	Instrument AVG400 Chemist c Group Cor

Uti... 1.34

Nature, 2021, s415...pdf | EN molecules-v26-i1...enw | molecules-26-0593...zip | Molecules, 2021, 2...pdf | manuscriptv7.docx | Fonr. Mol. Biosci, 2...pdf | EN 10.3389-fmolb.20...enw | Show all | X

# 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**
- ▶ Online booking (intranet)- registration is required:
- ▶ <https://nmr.chem.ox.ac.uk/hands>

# NMR Submission Service

- ▶ Many routine 1D and 2D  $^1\text{H}$ ,  $^{13}\text{C}$ ,  $^{19}\text{F}$ ,  $^{31}\text{P}$ , &  $^{11}\text{B}$  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  $^1\text{H}$  spectrum (PDF) *of same sample*

# NMR on-line Submission

## 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)

Here are the most recent submissions and their service status

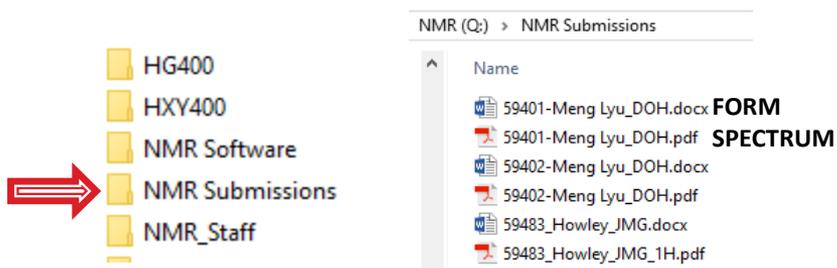
Date Submitted	Name	Research Group	Experiments Required	Sample Number	Completed	Instrument
02/10/2020	Lu Ying	SA	13C gHSQC gHMBC 29Si{1H} 77Se{1H} please	59520		
01/10/2020	anna vicini	VG	1H, 19F, 13C, HSQC VT (above rt), please and thank you	59519	Not yet	
01/10/2020	Joseph Ford	VG	1H, 19F, 13C, HSQC, COSY, HMBC please	59518		
01/10/2020	Victoria Atkinson	JR	13C, COSY, HSQC, HMBC please	59517		
01/10/2020	Katrina Andrews	SJC	1H, 13C, COSY, HSQC, HMBC please	59516		
01/10/2020	Stuart Astle	JB	1H, 13C, HSQC, HMBC, COSY Please	59515		
01/10/2020	Joseph Ford	VG	1H, 19F, 13C, HSQC, COSY, HMBC please	59514		
01/10/2020	Joseph Ford	VG	1H, 19F, 13C, HSQC, COSY, HMBC please	59513		

<https://web.chem.ox.ac.uk/samples/>

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)

## Part IIs should enter: p2

# Sample Submission Form: Word document



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

INTERNAL: NMR SERVICE SUBMISSION FORM FOR DEPARTMENT OF CHEMISTRY

**NMR@CHEM.OX** CHEMISTRY RESEARCH LABORATORY NMR SERVICE

Carousel #: Instr: Hours:

Name: Aidan Kerckhoffs Status: Post Doc CRL Lab: S8 CRL  
Email: aidan.kerckhoffs@chem.ox.ac.uk Phone: 75948  
Group: AJR [PI II's only] Lab. Supervisor's Name: 1

Submission Number: 2 71762 Project Code/ Charge Account: 3 DMR02040 Submission Date: 21/09/2023

Nuclei of interest: 1H, 13C Sample @: 4 **Rack** Fridge Request  
Structure: Toxicity: 5 unknown Solution Depths: 8  
Max 4.5 cm  
Min 4.0 cm

Experiments required (list ALL): 1H, 13C, COSY, HSQC, HMBC please

Nature of problem: 6  
Just need some spectra for characterisation! Thank you so much

Mass supplied: 7 10 mg Solvent: 8 DMSO

Referencing: 1H and 13C spectra are referenced externally to TMS in CDCl3, 19F spectra and 31P spectra are referenced externally to CFCl3 in CDCl3 and to phosphoric acid in D2O respectively. Indicate if you have added an internal reference.

- 1) Part II students must provide the name of their laboratory supervisor.
- 2) You should quote the number generated on the sample submission system page.
- 3) This is what you would use for iProcurement purchases. Seek advice from the Finance team if you do not know this.
- 4) Indicate where your sample can be found. If 'request', you will be contacted by the NMR staff in due course.
- 5) Give ANY details you may know that relate to possible hazards associated with handling of the sample (such as in the case of sample spillage or tube breakages). E.g. toxic, carcinogen etc. If this is uncertain, enter UNKNOWN.
- 6) Indicate the expected presence of unusual shifts. Describe briefly any particular problem you wish to address (this will help us choose the most appropriate experiment(s) for the problem). All experiments requested must be listed on this form.
- 7) 1H: 1-10 mg for the 600; 13C: 10+ mg for the 600 (ca. 50+ mg should be run on the 400s); 19F: 1-10mg; 31P: 10 mg. Please ask for others.
- 8) For routine analysis, all samples should be supplied in 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.
- 9) The maximum solvent depth for 5 mm tubes should be 4.5 cm (600µl), the minimum is 4.0 cm (500µl). Note that the automated spectrometers also require a sample depth of 4.0 - 4.5 cm. Samples with depths outside this range may be rejected.

# Online resources

- ▶ NMR web pages:
  - <https://nmr.chem.ox.ac.uk/>
  - or through **Intranet** link to **Facilities** then **Instrument booking and analytical services** on Chemistry homepage.

Login Department of Chemistry Instrument Booking System NMR Submission System



## NMR Facility

Chemistry Department NMR Research Facility



Home Services People Instruments Software Resources Research FAQs UKMRM



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 jointly by [Dr Nick Rees](#) and [Dr Harry Mackenzie](#), and is operated and maintained by four [members of staff](#) in total.

▲ Safety notice

# 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 online 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 2025: **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**

# Automated AVIIIHD400 [2<sup>nd</sup> 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 [2<sup>nd</sup> Floor]



Known as Venus400

- 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/venus400/setup.html>

# Hands on AVIII500 [basement]



Known as the AVD500

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

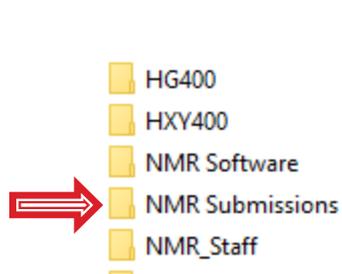
Booking required  
(online system)

# 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](mailto: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...*

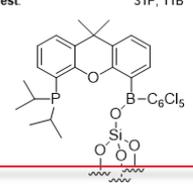
INTERNAL: NMR SERVICE SUBMISSION FORM FOR DEPARTMENT OF CHEMISTRY

**NMR@CHEM.OX** CHEMISTRY RESEARCH LABORATORY  
**NMR SERVICE**

Carousel #: \_\_\_\_\_  
Instr: \_\_\_\_\_  
Hours: \_\_\_\_\_

Name: Aisling Roper Status: D.Phil. CRL Lab: S12  
Email: aisling.roper@chem.ox.ac.uk Phone: \_\_\_\_\_  
Group: Aldridge [Pt II's only] Lab. Supervisor's Name:<sup>1</sup> \_\_\_\_\_  
Submission Number:<sup>2</sup> 70957 Project Code/ Charge Account:<sup>3</sup> DHT00110 DHSA.08 Submission Date: 06/07/23

Nuclei of interest: 31P, 11B **Sample @:<sup>4</sup> S2 glovebox – requires packing**

Structure:  Toxicity:<sup>5</sup> unknown Solution Depths:<sup>9</sup>  
Max 4.5 cm  
Min 4.0 cm

Experiments required (list ALL): solid state 31P and 11B NMR coupled and decoupled

Nature of problem:<sup>6</sup> expected 11B shift around +10-+60 ppm, expected 31P at approx. -10 ppm

Mass supplied:<sup>7</sup> \_\_\_\_\_ Solvent:<sup>8</sup> \_\_\_\_\_

Referencing: <sup>1</sup>H and <sup>13</sup>C spectra are referenced externally to TMS in CDCl<sub>3</sub>, <sup>19</sup>F spectra and <sup>31</sup>P spectra are referenced externally to CFC<sub>3</sub> in CDCl<sub>3</sub> and to phosphoric acid in D<sub>2</sub>O respectively. Indicate if you have added an internal reference.

- 1) Part II students must provide the name of their laboratory supervisor.
- 2) You should quote the number generated on the sample submission system page.
- 3) This is what you would use for iProcurement purchases. Seek advice from the Finance team if you do not know this.
- 4) Indicate where your sample can be found. If 'request', you will be contacted by the NMR staff in due course.
- 5) Give ANY details you may know that relate to possible hazards associated with handling of the sample (such as in the case of sample spillage or tube breakages). E.g. toxic, carcinogen etc. If this is uncertain, enter UNKNOWN.
- 6) Indicate the expected presence of unusual shifts. Describe briefly any particular problem you wish to address (this will help us choose the most appropriate experiment(s) for the problem). All experiments requested must be listed on this form.
- 7) <sup>1</sup>H: 1-10 mg for the 600; <sup>13</sup>C: 10+ mg for the 600 (ca. 50+ mg should be run on the 400s); <sup>19</sup>F: 1-10mg; <sup>31</sup>P: 10 mg. Please ask for others.
- 8) For routine analysis, all samples should be supplied in 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.
- 9) The maximum solvent depth for 5 mm tubes should be 4.5 cm (600µl), the minimum is 4.0 cm (500µl). Note that the automated spectrometers also require a sample depth of 4.0 - 4.5 cm. Samples with depths outside this range may be rejected.

# Solid State AVIIIHD400WB [basement]

Known as the HXY400



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

# Solid State AVIIIHD400WB [basement]



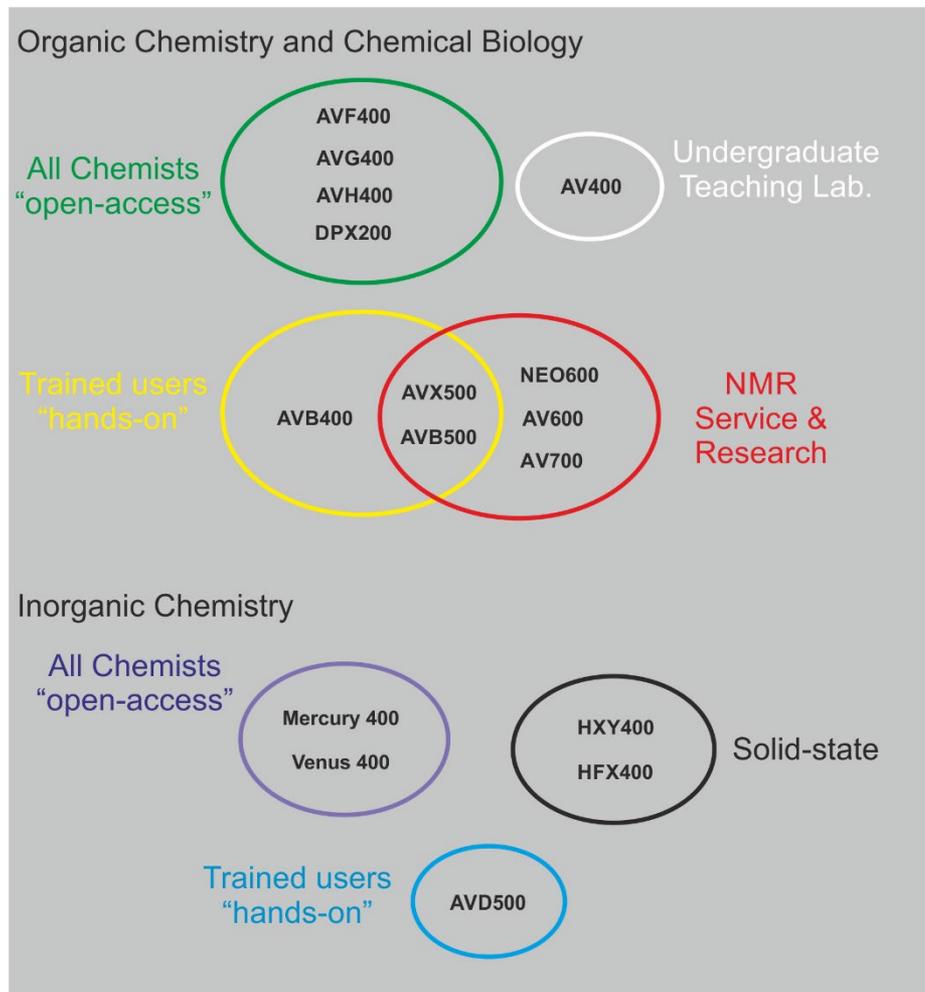
Known as the HFX400

- 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 150C)

# 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.*
  - Sessions will run Wednesday 2<sup>nd</sup> (pm), Thurs 3<sup>rd</sup> (pm) and Fri 4<sup>th</sup> (am): **Meet in CRL reception**
  - **Online booking and registration is required**
- ▶ Mnova NMR Software Introductory Lecture
  - Single on-line lecture introducing main software features
- ▶ CDT students
  - *NMR training courses in Jan 2025: **DO NOT SIGN UP FOR TRAINING THIS WEEK***

# Instrumentation Access



# 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:**

<https://outlook.office365.com/owa/calendar/SRFInductions2024@UniOxfordNexus.onmicrosoft.com/bookings/s/bgg8DtMOmEmNP7fT42ZQhQ2>

or **Inorganic Section NMR:**

[https://outlook.office365.com/owa/calendar/SRFInductions2024@UniOxfordNexus.onmicrosoft.com/bookings/s/Cf0I\\_LTuLk2LQKJEyn2onw2](https://outlook.office365.com/owa/calendar/SRFInductions2024@UniOxfordNexus.onmicrosoft.com/bookings/s/Cf0I_LTuLk2LQKJEyn2onw2)

**Meet for training in CRL reception**

To arrange training external users should email: [nmrstaff@maillist.chem.ox.ac.uk](mailto:nmrstaff@maillist.chem.ox.ac.uk)

All the above links can be found at:

<https://massspec.chem.ox.ac.uk/book>

# QR for training sign-up sheets

