



Chemistry Research Laboratory

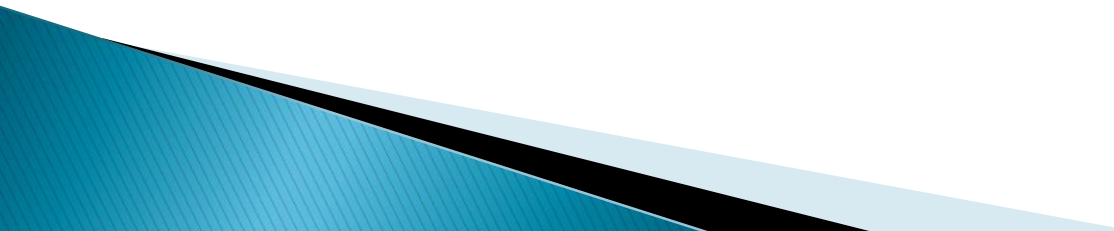
# NMR Spectroscopy Facility Introductory Lecture

Dr. Nick Rees

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[nmrweb.chem.ox.ac.uk](http://nmrweb.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
  - ▶ 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](mailto:nmrstaff@maillist.chem.ox.ac.uk)



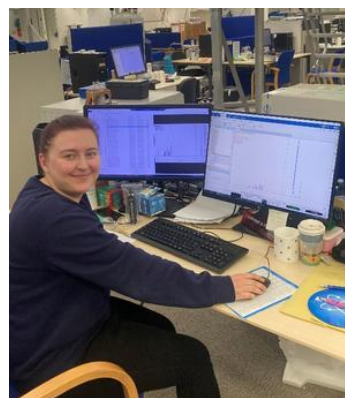
Nick



Caitlin



James



Coral

# 1. NMR Staff

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

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



Nick



Caitlin



Coral



Charlie

# 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



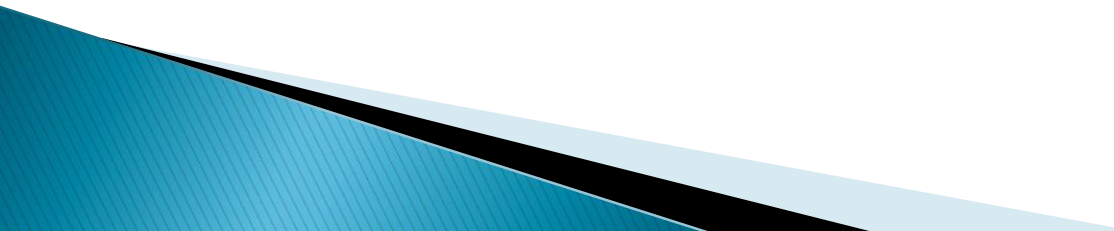
- ▶ No laboratory coats in NMR labs.
- ▶ No metal objects to be taken into NMR labs.
- ▶ Sample breakages must be dealt with immediately
  - Inform the NMR staff if in any doubt

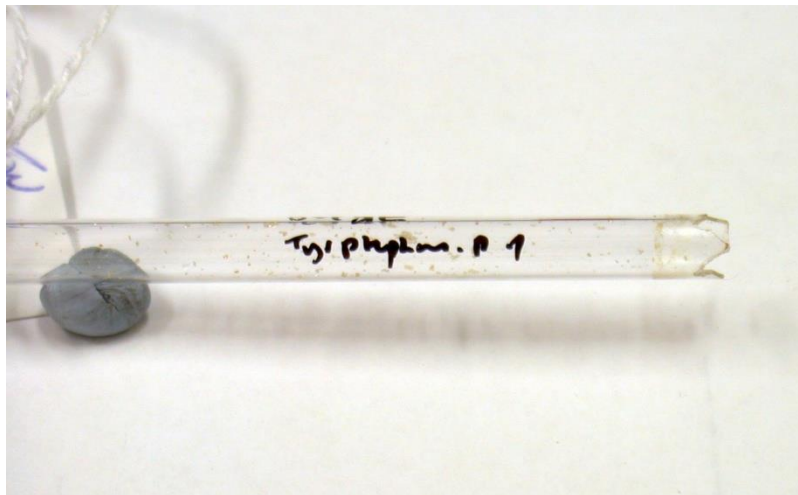
# Accessibility in the NMR laboratories

- ▶ 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)



### 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

- ▶ 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 Tube Cap Colours for Organic Chemistry and Chemical Biology groups

Group	Floor	Colour
Professor E. A. Anderson	1	WHITE
Professor J. Burton	1	YELLOW
Professor T. J. Donohoe	1	PURPLE
Professor V. Gouverneur	1	PINK + BLACK
Professor D. M. Hodgson	1	FUCHSIA + BLACK
Professor M. G. Moloney	1	GREEN + BLACK
Professor M. D. Smith	1	BLUE + BLACK
Professor M. C. Willis	1	AQUA + BLACK
Professor H. L. Anderson	g	SKY
Professor S. J. Conway	g	AQUA
Professor S. G. Davies	g	RED
Professor D. J. Dixon	g	BLACK
Professor S. P. Fletcher	g	SKY + BLACK
Professor I. McCulloch	g	RED
Professor J. Robertson	g	ORANGE + BLACK
Professor A. J. Russell	g	FUCHSIA
Professor H. Bayley	lg	PINK
Dr. M. J. Booth	lg	WHITE + BLACK
Professor B. G. Davis	lg	GREEN
Professor C. J. Schofield	lg	ORANGE
Professor T. Brown		BLUE
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  $\text{CuSO}_4$



# 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>

Name	Name	Name
AV600	2012	ajrgp
AV700	2013	bgdgrp
AVB400	2014	cjsgrp
AVB500	2015	djdgrp
AVC500	2016	dmhgrp
AVD500	2017	eaagrp
AVF400	2018	hbgrp
AVG400		hlagrp
AVH400		jbgrp
AVX500		jrgrp
cmx200		lsfgrp
cmx400		mcwgrp
DPX200		mdsgrp
DPX250		memgrp
DPX300		
DPX400		
DQX400		
DRX500		
HFX400		
hg300-crl		
hg400		
HXY400		

Name
Sep30-2018-29-PKD 360 7-14
Sep30-2018-45-PKD 360 45-58
Sep30-2018-45-PKD 360 33-44
Sep30-2018-30-PKD 360 crude
475 B
475 A
474 A PRODUCT



# 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



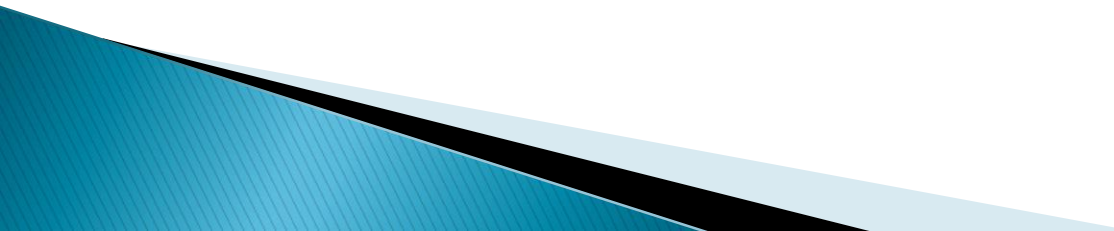


# Software installation

## ▶ 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

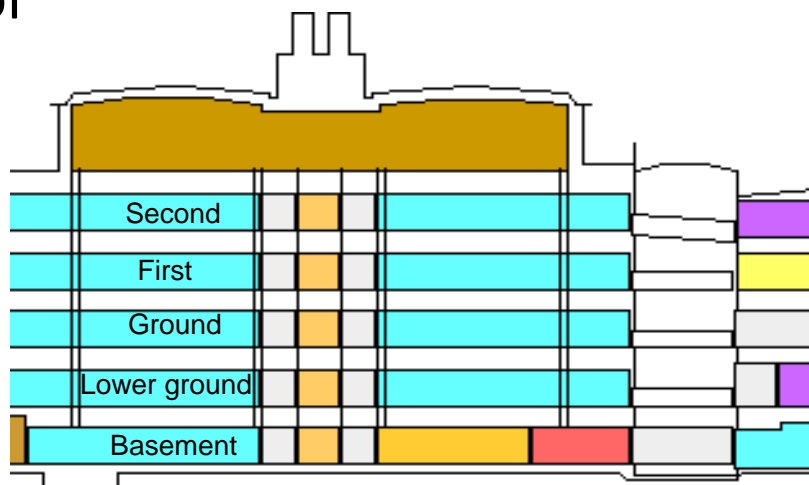
[Instrument nicknames shown]

## 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
  - 1 @ 700 MHz: Research projects

# 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 Tuesday 3<sup>rd</sup> (pm), Thurs 5<sup>th</sup> (pm) and Fri 6<sup>th</sup> (am): **Meet in CRL reception**
  - **On-line 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

HOLDER	STATUS	NAME	EXP NO / EXPERIMENT	USER	TIME	TITLE
1	Completed	Sep30-2021-1-DH_03_155_hexyl-dry	1 N hlaqc.crl 1H	mjbgrp	00:01:32	Instrument AVG400 Chemist DH Group MJB Project Account Code DMT00010
2	Completed	NB008	2 N hlaqc.crl 1H	mjbgrp	00:01:32	Instrument AVG400 Chemist NB Group MJB Project Account Code DQR00150 NB008 recrystallised
3	Completed	BF130-14-19	1 N hlaqc.crl 1H	djdgrp	00:01:32	Instrument AVG400 Chemist BF Group DJD Project Account Code other
3	Completed	BF130-14-19	2 N p31dec.crl Phosphorus with 1H decoupling +200 to -200 ppm	djdgrp	00:01:01	Instrument AVG400 Chemist BF Group DJD Project Account Code other
4	Completed	Sep30-2021-4-MLJM-B16	1 N hlaqc.crl 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 hlaqc.crl 1H	hlagrp	00:01:32	Instrument AVG400 Chemist TT Group HLA Project Account Code Other
7	Completed	Sep30-2021-7-OJS84_(1)_2	1 N hlaqc.crl 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 hlaqc.crl 1H	djdgrp	00:01:32	Instrument AVG400 Chemist Tanya Group DJD Project Account Code other
8	Completed	Sep30-2021-8-RT-9-867-pure	2 n c13aqc_512.crl Carbon 512 scans	djdgrp	00:14:33	Instrument AVG400 Chemist Tanya Group DJD Project Account Code other
9	Completed	Sep30-2021-9-SM-5-93_crd	1 N hlaqc.crl 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 hlaqc.crl 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 hlaqc.crl 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 hlaqc.crl 1H	cjsgrp	00:01:32	Instrument AVG400 Chemist RT Group CJS Project Account Code PT2
13	Completed	mm287	1 N hlaqc.crl 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-akl-690-3-crude1	1 N hlaqc.crl 1H	ajrgp	00:01:32	Instrument AVG400 Chemist ak Group AJR Project Account Code DM8000
16	Completed	Sep30-2021-16-3332-novo	1 N hlaqc.crl 1H	ajrgp	00:01:32	Instrument AVG400 Chemist cjrb Group AJR Project Account Code DM8000 cjrb3332-novo
16	Completed	Sep30-2021-16-3332-novo	2 N fl9aqc.crl Fluorine +100 to -250 ppm (16 scans)	ajrgp	00:01:00	Instrument AVG400 Chemist cjrb Group AJR Project Account Code DM8000 cjrb3332-novo
16	Submitted	Sep30-2021-16-3332-novo	3 n c13aqc_512.crl Carbon 512 scans	ajrgp	00:14:33	Instrument AVG400 Chemist cjrb Group AJR Project Account Code DM8000 cjrb3332-novo
16	Submitted	Sep30-2021-16-3332-novo	n DEPT135.crl AV400 13C DEPT135	ajrgp	00:03:41	Instrument AVG400 Chemist cjrb Group AJR Project Account Code DM8000 cjrb3332-novo
17	Completed	Sep30-2021-17-CA-2-071	1 N hlaqc.crl 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 hlaqc.crl 1H	imgrp	00:01:32	Instrument AVG400 Chemist C Aitchison Group IM Project Account Code DMR01750
19	Completed	FM 07-61 AC	1 N hlaqc.crl 1H	spfgrp	00:01:32	Instrument AVG400 Chemist FM Group SPF Project Account Code DMR00790
20	Completed	Sep30-2021-20-ALR01	1 N hlaqc.crl 1H	ajrgp	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.crl	jrgp	00:05:05	Instrument AVG400 Chemist SL Group JR Project Account Code dm7900 fr 2
22	Completed	IM-001	1 N hlaqc.crl 1H	djdgrp	00:01:32	Instrument AVG400 Chemist Iain McLauchlan Group DJD Project Account Code other
22	Submitted	IM-001	2 n c13aqc_512.crl Carbon 512 scans	djdgrp	00:14:33	Instrument AVG400 Chemist Iain McLauchlan Group DJD Project Account Code other
22	Completed	IM-001	3 n fl9dec.crl Fluorine with 1H decoupling +100 to -250 ppm (16 scans)	djdgrp	00:01:00	Instrument AVG400 Chemist Iain McLauchlan Group DJD Project Account Code other
22	Submitted	IM-001	4 n COSY.crl	djdgrp	00:05:05	Instrument AVG400 Chemist Iain McLauchlan Group DJD Project Account Code other
22	Submitted	IM-001	5 n HSQC.crl	djdgrp	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.crl Phosphorus with 1H decoupling +200 to -200 ppm	bvlpgp	00:01:01	Instrument AVG400 Chemist mls Group BVLP
24	Completed	Sep30-2021-24-468x2-10-12	1 N p31dec.crl Phosphorus with 1H decoupling +200 to -200 ppm	bvlpgp	00:01:01	Instrument AVG400 Chemist mls Group BVLP
25	Completed	Sep30-2021-25-468x2-13-15	1 N hlaqc.crl 1H	bvlpgp	00:01:32	Instrument AVG400 Chemist mls Group BVLP
25	Running	Sep30-2021-25-468x2-13-15	2 N p31dec.crl Phosphorus with 1H decoupling +200 to -200 ppm	bvlpgp	00:01:01	Instrument AVG400 Chemist mls Group BVLP
26	Submitted	DSCH_04_019_col2_3_f22-28	1 N hlaqc.crl 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 hlaqc.crl 1H	hlagrp		Instrument AVG400 Group HLA
29	Submitted	Sep30-2021-29-468x2-16-18	1 N hlaqc.crl 1H	bvlpgp	00:01:32	Instrument AVG400 Chemist mls Group BVLP
29	Submitted	Sep30-2021-29-468x2-16-18	2 N p31dec.crl Phosphorus with 1H decoupling +200 to -200 ppm	bvlpgp	00:01:01	Instrument AVG400 Chemist mls Group BVLP
39	Error	Sep29-2021-38-2066SM meod	1 N hlsup.crl 1H presat	corgp	00:01:32	Instrument AVG400 Chemist c Group Cor

# 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>

# 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		

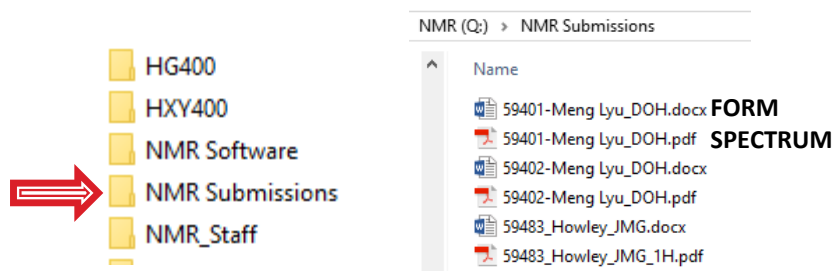
<http://www.chem.ox.ac.uk/samples>

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 [Pt II's only] Lab. Supervisor's Name:

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

Nuclei of interest: <sup>1</sup>H, <sup>13</sup>C Sample @:<sup>4</sup> Rack Fridge Request

Structure: Toxicity: <sup>5</sup> unknown Solution Depths:<sup>8</sup>

Max 4.5 cm  
Min 4.0 cm

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

Nature of problem:<sup>6</sup>

Just need some spectra for characterisation! Thank you so much

Mass supplied:<sup>7</sup> 10 mg Solvent:<sup>8</sup> DMSO

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 CFCl<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.

# On-line resources

- ▶ NMR web pages:

- <http://nmrweb.chem.ox.ac.uk/>
- or through **Intranet** Link to **Analytical Facilities** on Chemistry homepage.

The screenshot shows a web browser displaying the 'NMR Facility' website. The browser's address bar shows 'https://wessington.chem.ox.ac.uk/Default.aspx'. The website header includes the 'DEPARTMENT OF CHEMISTRY' and 'UNIVERSITY OF OXFORD' logos, and the title 'NMR Facility'. A navigation menu contains links: 'STUDY HERE', 'WORKING HERE', 'RESEARCH', 'PEOPLE', 'OUR STUDENTS', and 'ABOUT'. Below this is a secondary menu with 'HOME', 'STAFF', 'INSTRUMENTS', 'SOFTWARE', 'RESOURCES', and 'RESEARCH'. The left sidebar lists links: 'How to use', 'NMR Services', 'Sample Submission System', 'Instrument Booking System', and 'History lists'. The main content area features a large photo of the NMR facility, followed by the heading 'NMR Operations following reduced COVID restrictions'. The text states that the department will retain new protocols for sample submissions and provides contact information for NMR staff. It also describes the facility's capabilities, including thirteen solution-state and two solid-state FT NMR instruments with proton operating frequencies up to 700 MHz. The facility supports a full range of chemical sciences research, including synthetic organic and inorganic chemistry, supramolecular chemistry, chemical biology, enzymology, metabolomics, catalysis, and materials science. The facility is managed by Prof Tim Claridge (Organic/ChemBiol) and Dr Nick Rees (Inorganic) and is operated and maintained by five members of staff in total. A 'Latest News' section mentions a 2021 introductory lecture and a new handout on Diffusion NMR and DOSY.

DEPARTMENT OF CHEMISTRY  
UNIVERSITY OF OXFORD

NMR Facility

STUDY HERE | WORKING HERE | RESEARCH | PEOPLE | OUR STUDENTS | ABOUT

HOME | STAFF | INSTRUMENTS | SOFTWARE | RESOURCES | RESEARCH

How to use  
NMR Services  
Sample Submission System  
Instrument Booking System  
History lists

Commercial NMR Services

The department also offers external NMR services for industry.  
Details may be found on our [Commercial Services](#) page.

Welcome

**NMR Operations following reduced COVID restrictions**

We shall retain our [new protocols for sample submissions](#) to the NMR Service.

Contact [NMR staff](#) if you require use of a basement instrument, explaining your need, so that appropriate training can be arranged.

The NMR facility housed in the Chemistry Research Laboratory 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.

They are managed by [Prof Tim Claridge](#) (Organic/ChemBiol) and [Dr Nick Rees](#) (Inorganic) and are operated and maintained by five members of staff in total.

**Latest News:**

**2021 Introductory lecture:** The lecture slides will be found [here](#) after the introductory lectures on 5th October (Oxford only).

**20/4/20:** We have a new handout on Diffusion NMR and DOSY: Available from our [Resources-NMR Handouts](#) page

Nature, 2021, s415...pdf | molecules-26-v11...enw | molecules-26-0593...zip | Molecules, 2021, 2...pdf | manuscriptv7.docx | For. Mol. Biosci, 2...pdf | 10.3389-fmolb.20...enw | Show all

# 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**  $^1\text{H}$  and  $^{13}\text{C}$

# 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]

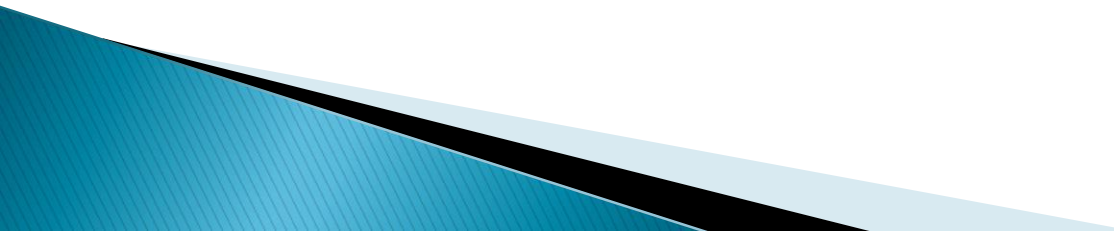


Known as the AVD500

- 24 place autosampler
- $^1\text{H}$ ,  $^{19}\text{F}$  to  $^{109}\text{Ag}$
- $^1\text{H}$ – $^1\text{H}$ ,  $^1\text{H}$ -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](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


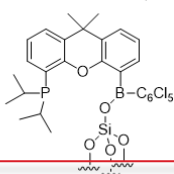


- HG400
- HXY400
- NMR Software
- NMR Submissions
- NMR\_Staff

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			
		<b>CHEMISTRY RESEARCH LABORATORY</b> <b>NMR SERVICE</b>	
Name: Aisling Roper		Status: D.Phil.	Carousel #: _____ Instr: _____ Hours: _____
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> _____	
<p>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 CFCl<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.</p> <p>1) Part II students must provide the name of their laboratory supervisor.</p> <p>2) You should quote the number generated on the sample submission system page.</p> <p>3) This is what you would use for iProcurement purchases. Seek advice from the Finance team if you do not know this.</p> <p>4) Indicate where your sample can be found. If 'request', you will be contacted by the NMR staff in due course.</p> <p>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.</p> <p>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.</p> <p>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.</p> <p>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.</p> <p>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.</p>			



# 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

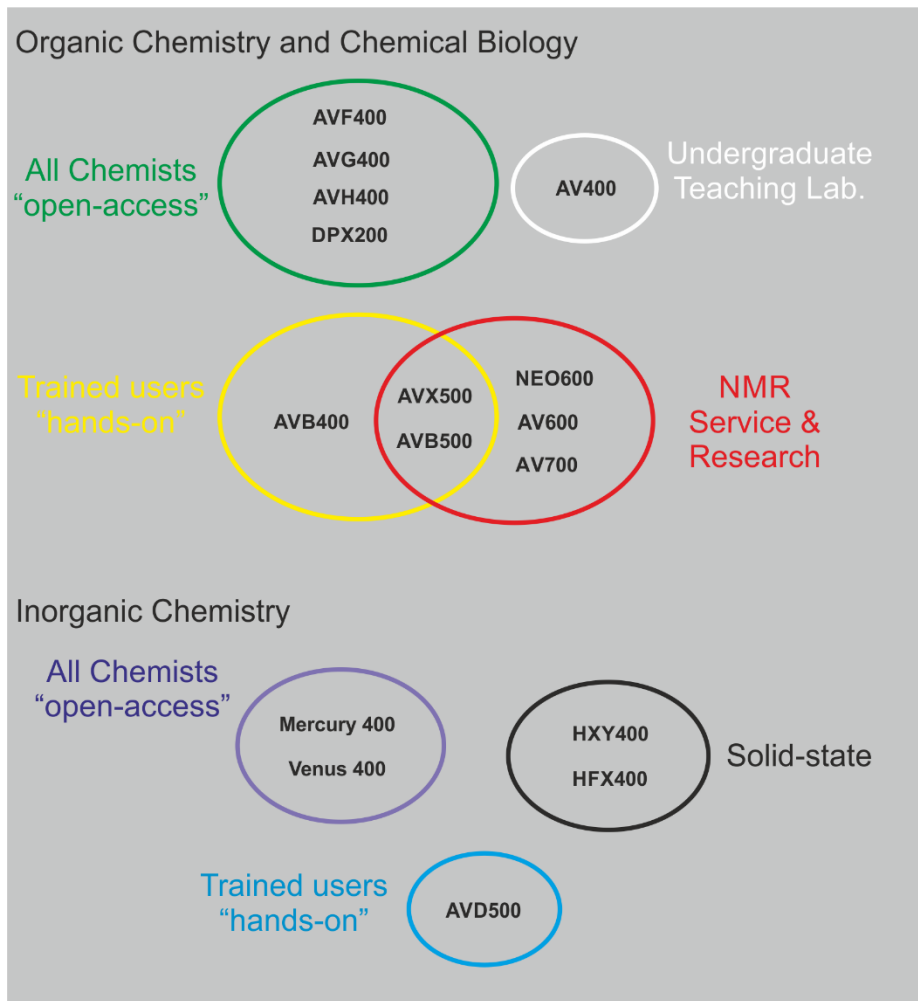
- 3.2mm HFX Triple Magic Angle Spinning Probes ( $^1\text{H}$ ,  $^{19}\text{F}$ ,  $^{31}\text{P}$  to  $^{15}\text{N}$ )
- 30mm Micro-imaging probe
- Diffusion probe
- Variable temperature capable (-150 to 150C)



# 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.***
- ▶ 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



# 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](mailto: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  $^1\text{H}$  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  $^1\text{H}$  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:  $^2\text{H}$ ,  $^7\text{Li}$ ,  $^{27}\text{Al}$
- 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  $\mu$ L)

## Quantity of sample

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

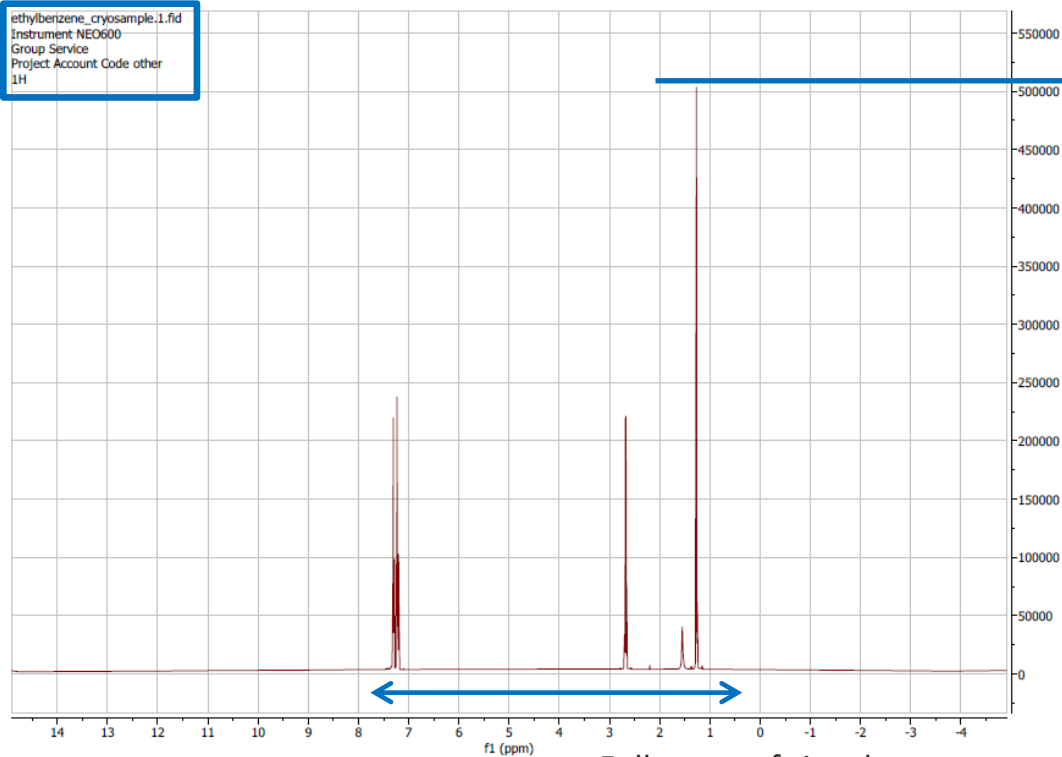
## Labelling of NMR tube

- Use the correct NMR cap colour
- Label cap/tube with initials, solvent and group

## Step 2: Acquire A $^1\text{H}$ NMR And Convert Spectrum To A PDF.

Spectrum  
information

ethylbenzene\_cryosample.1.fid  
Instrument NEO600  
Group Service  
Project Account Code other  
1H



Top of the most  
intense signal  
can be seen

# 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>



# NMR Facility

Chemistry Department NMR Research Facility



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The NMR facility is managed by [Dr Nick Rees](#) 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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Chemistry Department NMR Research Facility

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Open-access

Submission Service

Hands-on



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## 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](#).

[+ Expand All](#)

Internal samples (i.e. researchers in Chemistry)



External samples (i.e. researchers from other departments)



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[Accessibility statement](#)  
[Privacy statement](#)



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Chemistry Department NMR Research Facility



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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 ID  $^1\text{H}$  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.

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)

Here are the most recent submissions and their service status

Date Submitted	Name	Research Group	Experiments Required	Sample Number	Completed	Instrument
28/9/2023	Maddie Hindson	MDS	1H, 13C, COSY, HMBC, HSQC pls	71800		
28/9/2023	Maddie Hindson	MDS	1H, 13C, COSY, HMBC, HSQC pls	71799		
28/9/2023	Maddie Hindson	MDS	1H, 13C, COSY, HMBC, HSQC pls	71798		
28/9/2023	Zhuxin Zhang	CJS	1H, 13C, HMBC please	71797		
27/9/2023	Marta Serafini	SJC	1H, 13C, COSY, HSQC, HMBC please	71796		
27/9/2023	Nicola L...	IB	1H, 13C, COSY, HSQC, HMBC	71795		

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27/9/2023	Nicola L...	...	1H, 13C, COSY, HSQC, HMBC	71795		

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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
28/9/2023	Coral Mycroft	OTHER	1H, 13C, COSY, HSQC	71801		
28/9/2023	Maddie Hindson	MDS	1H, 13C, COSY, HMBC, HSQC pls	71800		
28/9/2023	Maddie Hindson	MDS	1H, 13C, COSY, HMBC, HSQC pls	71799		
28/9/2023	Maddie Hindson	MDS	1H, 13C, COSY, HMBC, HSQC pls	71798		
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+ Expand All

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

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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 <sup>1</sup>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.  
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AutoSave nmrserviceforminternal2021.docx(3).docx - Word Coral Mycroft CM

File Home Insert Design Layout References Mailings Review View Help Nuance PDF

Clipboard Font Paragraph Styles Editing

INTERNAL: NMR SERVICE SUBMISSION FORM FOR DEPARTMENT OF CHEMISTRY

**CHEMISTRY RESEARCH LABORATORY**  
**NMR SERVICE**

Carousel #: Instr: Hours:

Name:	Status: Pt II D.Phil. Post Doc	CRL Lab:
Email:		Phone:
Group:	[Pt II's only] Lab. Supervisor's Name: <sup>1</sup>	
Submission Number: <sup>2</sup>	Project Code/ Charge Account: <sup>3</sup>	Submission Date:

Nuclei of interest: Sample @:<sup>4</sup> Rack Fridge Request

Structure: Toxicity:<sup>5</sup> Solution Depths:<sup>6</sup>

Max 4.5 cm  
Min 4.0 cm

Experiments required (list ALL):

Nature of problem:<sup>7</sup>

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 CFCl<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.
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- 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 submitted in 5 mm high quality tubes (Borall ADNS 300000 507 DD or NMR FRS MDC at least).

Page 1 of 1 353 words

inbox - coral.mycroft... Re: NMR training link... Submission service | ... nmrserviceformi... Documents Mail - Coral Mycroft ... NMR Service - Open ...

Personal information

Sample and experiment information (mostly)

Guidance on filling out the submission form

## INTERNAL: NMR SERVICE SUBMISSION FORM FOR DEPARTMENT OF CHEMISTRY

NMR@CHEM.OX

## CHEMISTRY RESEARCH LABORATORY

## NMR SERVICE

Carousel #:

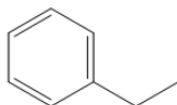
Instr:

Hours:

Name: Coral Mycroft	Status: <b>Pt II</b> D.Phil. Post Doc	CRL Lab: <b>B00.120</b>
Email: <a href="mailto:nmrstaff@maillist.chem.ox.ac.uk">nmrstaff@maillist.chem.ox.ac.uk</a>		Phone: <b>12345</b>
Group: <b>VG / CJS / HLA / DOH</b>	[Pt II's only] Lab. Supervisor's Name: <b>D.Phil/Post Doc name</b>	
Submission Number: <sup>2</sup> <b>71801</b>	Project Code/ Charge Account: <sup>3</sup> <b>DMT12345</b>	Submission Date: <b>28/09/2023</b>

Nuclei of interest: **<sup>1</sup>H, <sup>13</sup>C**Sample @:<sup>4</sup> **Rack** Fridge Request

Structure:

Toxicity: <sup>5</sup> **FLAMMABLE. HARMFUL.**Solution Depths:<sup>9</sup>

Max 4.5 cm

Min 4.0 cm

Experiments required (list ALL):

**<sup>1</sup>H, <sup>13</sup>C, COSY, HSQC**Nature of problem:<sup>6</sup>Standard structure assignment of new compound. Sample too dilute for <sup>13</sup>C and HSQC on open access spectrometers.Mass supplied:<sup>7</sup> **< 1 mg**Solvent:<sup>8</sup> **CDCl<sub>3</sub>**

# Step 5: Place Completed Form And PDF In The NMR Submissions Folder In The NMR Data Server

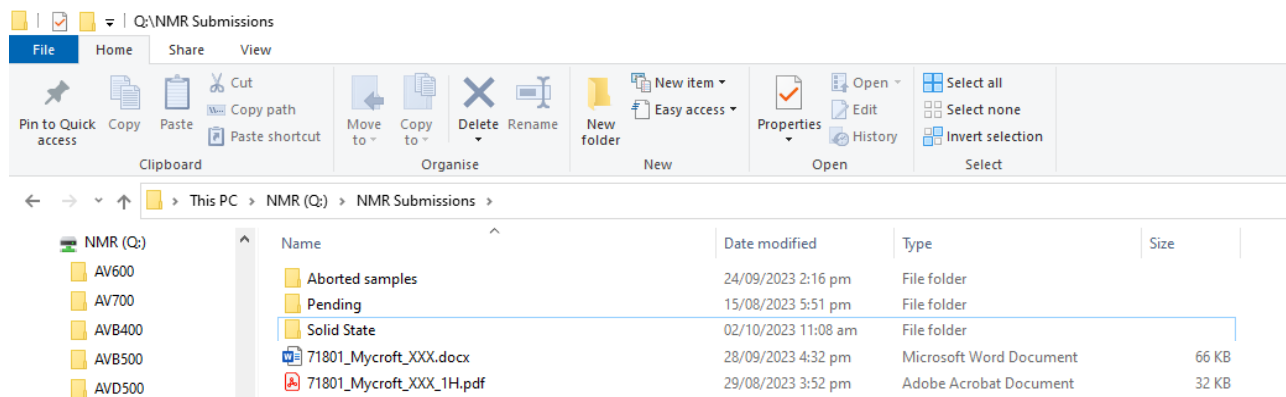
**Word:** <submission number>\_<name>\_<supervisor initials>

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.





# NMR Facility

Chemistry Department NMR Research Facility



[Home](#) **[Services](#)** [People](#) [Instruments](#) [Software](#) [Resources](#) [Research](#) [FAQs](#) [UKMRM](#)

## 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](#).

[+ Expand All](#)

### 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  $^1\text{H}$  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.

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 Facility

Chemistry Department NMR Research Facility



Home Services ▾ People Instruments ▾ **Software** Resources ▾ Research ▾ FAQs UKMRM

## 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.

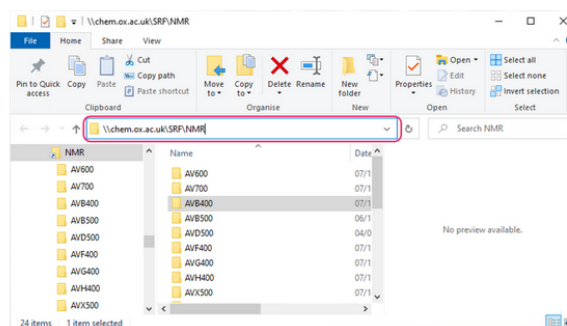
- **Open-access:** this is automatically done
- **Submission service:** this is automatically done, please email [nmrstaff@maillist.chem.ox.ac.uk](mailto:nmrstaff@maillist.chem.ox.ac.uk) if you cannot find your data
- **Hands-on:** this is automatically done if you ran your experiments under IconNMR 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)



# NMR Facility

Chemistry Department NMR Research Facility



[Home](#) **Services** [People](#) [Instruments ▼](#) [Software ▼](#) [Resources ▼](#) [Research ▼](#) [FAQs](#) [UKMRM](#)

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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>.**  
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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.

# Step 6: Bring The NMR Sample Down To The Basement With A Completed Label

Solution-state incoming and completed



Solid state incoming and completed



# Step 6: Bring The NMR Sample Down To The Basement With A Completed Label

NMR Spectroscopy	SAMPLE NUMBER:	71801	HOLDER:	FOR NMR SERVICE ONLY – LEAVE BLANK
	NAME:	Coral Mycroft		
	GROUP:	HLA/ HB/ SJC	DATE:	28/09/2023
	COST CODE:	DMT12345		
	SOLVENT:	CDCl <sub>3</sub>	STORAGE (RACK / FRIDGE):	Rack
	EXPERIMENTS:	<sup>1</sup> H, <sup>13</sup> C, COSY, HSQC		

# 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

Date Submitted	Name	Research Group	Experiments Required	Sample Number	Completed	Instrument
28/9/2023	Coral Mycroft	OTHER	1H, 13C, COSY, HSQC	71801	Yes	AVX500

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

# NMR Facility Members

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

Nick



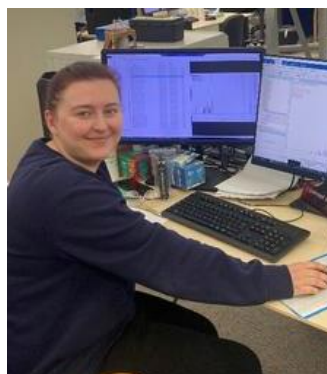
Head of NMR

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