



# NMR Facility Introductory Lecture

Dr. Nick Rees

Dr. Harry Mackenzie





#### Outline

**NMR Facility Staff** 

**Magnet Hazards and Safety** 

**Sample Preparation** 

**Data Processing** 

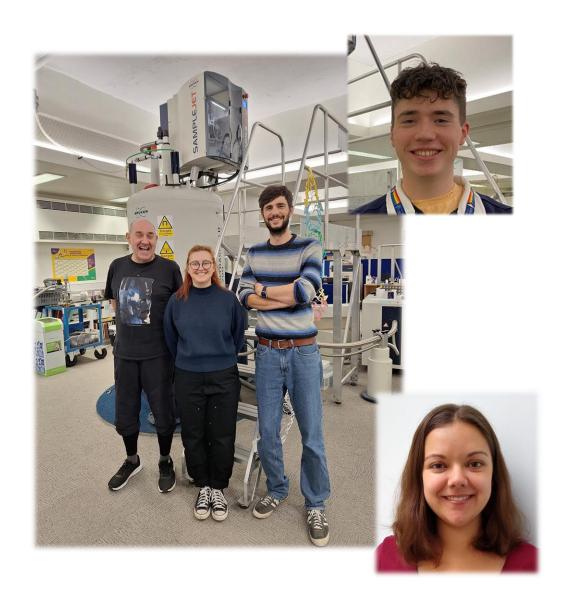
**Facilities and Instrumentation** 

Open Access Facilities

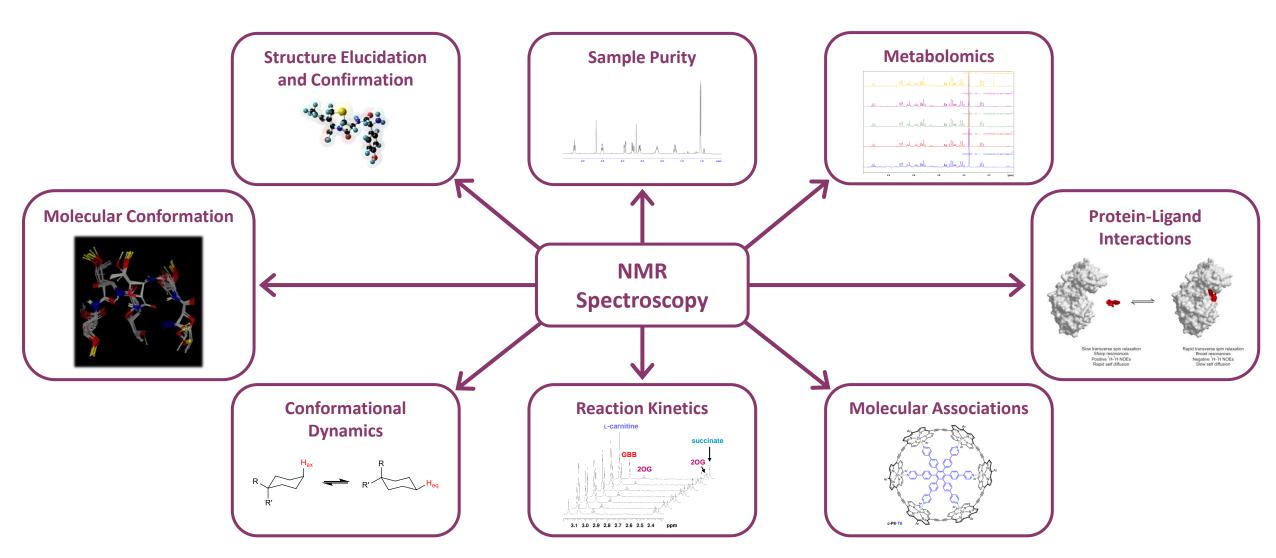
NMR Submission Service

Online Resources: NMR web site

**Future Training Courses** 











### **Facility Staff**

**Dr. Nick Rees** 

Head of Inorganic and Solid-State NMR

**Dr. Harry Mackenzie** 

Head of Organic and Biological NMR

**Dr. Coral Mycroft** 

Service Manager

**Caitlin Salter Edward Tomlinson (Maternity)** 

Service Technician













### Safety in the NMR Laboratories

**Very Strong** Magnetic Fields!

#### Hazards to:

heart pacemakers magnetic bank, ID cards watches (non-LCD)

Stray fields in corridors!

especially ground floor NMR





Cryogenic Liquids: extreme cold, oxygen depletion





### Safety Rules

**Clean Spaces**: No laboratory coats or gloves in NMR labs.

No metal objects to be taken into NMR labs.



Sample breakages must be dealt with <u>immediately</u>

Inform the NMR staff in all cases: nmrstaff@maillist.chem.ox.ac.uk



**Do not enter** labs during weekly liquid nitrogen re-fills





### 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

or

harry.mackenzie@chem.ox.ac.uk





### 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





### Sample Preparation: Bad Practice







### 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 labelled with the surname initial of your PI

Your own name/initials must be labelled on the tube

The **solvent** used must be labelled on the tube

**NOTE**: 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 information





### Cap Colours: Organic and Chemical Biology

- ( **A** ) E. Anderson
- A H. Anderson
- A. Avestro
- A Y. Aye
- **B** H. Bayley
- **B** J. Burton
- **(c)** D. Congrave

- D. Dixon
- A T. Donohoe
- **F** S. Fletcher
- M. Fuchter
- **G** V. Gouverneur
- M I. McCulloch
- M P. McGonigal

- **Q** Y. Qing
- R P. Rabe
- R J. Robertson
- R A. Russell
- S C. Schofield
- M. Smith
- W M. Willis





### Cap Colours: Inorganic Chemistry

- A S. Aldridge
- B P. Beer
- J. Davis
- S. Faulkner
- **G** G. Gregory
- (H) R. Hoye
- (L) M. Langton

- M. Mehta
- N M. Neidig
- D. O'Hare
- T E. Tsang
- K. Vincent
- **W** C. Williams
- W L. Wong

- N NMR
- X External





### Sample Masses Required

Rule of thumb for high-quality spectra

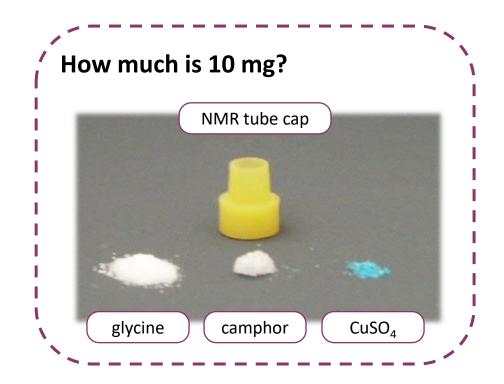
400 MHz open-access spectrometers:

Proton & 2D COSY: 2 mg

2D H-C HSQC: **10 mg** 

1D Carbon: **20 mg** 

Please weigh your NMR samples!!







### **Data Processing**

Data from all spectrometers can **ONLY** be downloaded from the SRF server for offline processing and local storage

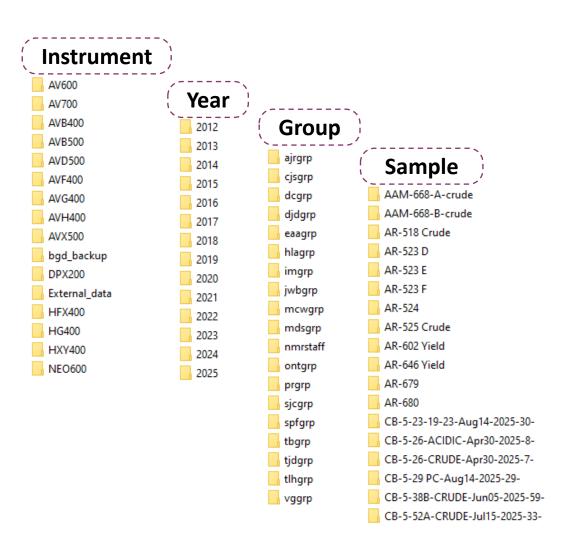
#### Windows:

\\chem.ox.ac.uk\srf\nmr

#### macOS:

smb://chem.ox.ac.uk/srf/nmr

**PLEASE** no USB drives







### **Data Processing Software**

Windows and macOS:

MestreNova (MestreLab):

Platform independent, 1D and 2D processing Chemistry site licence



Windows and macOS:

TopSpin (Bruker):

used on all spectrometers, 1D and 2D processing free for academic use







#### Software Installation

#### MestreNova 16.0:

Download from the Mestrelab website: <a href="https://mestrelab.com/download">https://mestrelab.com/download</a>

Drag and drop licence from NMR server: /NMR Software/Mnova/

#### TopSpin 4.5:

Download from Bruker website and request licence:

https://store.bruker.com/en-GB/products/topspin-for-processing-academic-government/





### NMR Facility – 4 Levels Of Analysis

#### **Open Access**

Walk up use at any time of the day

Common 1D/2D experiments: <sup>1</sup>H, <sup>13</sup>C, <sup>19</sup>F, <sup>31</sup>P, DEPT, COSY, HSQC, HMBC\*, NOESY\*

Fast turnover and hence rapid sample throughput

Ground/first floors: Organic Chemistry and Chemical Biology

Second floor: Inorganic Chemistry

#### Hands on

Users can book the basement systems for manual/automated operation

#### **NMR Service**

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

#### **Research Projects**

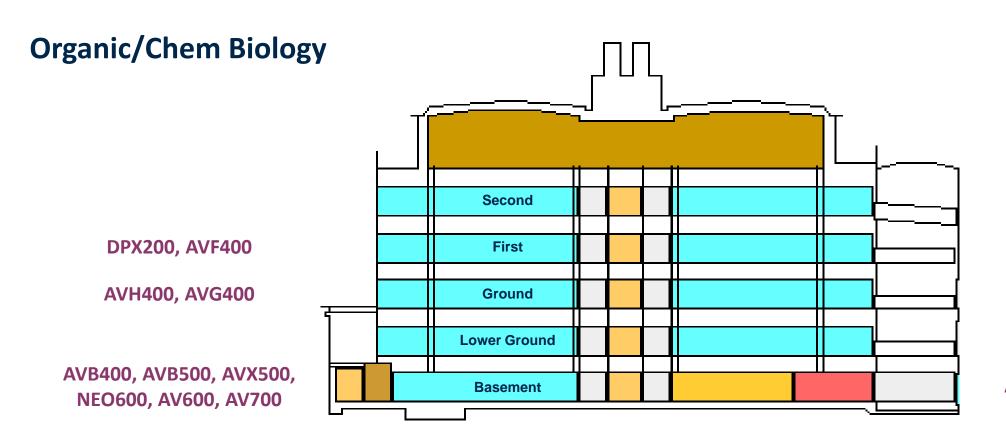
Collaborative projects involving the NMR staff/group





#### NMR in CRL

[Instrument nicknames shown]



#### **Inorganic Section**

Venus400, Hg400

AVD500, HFX400, HXY400

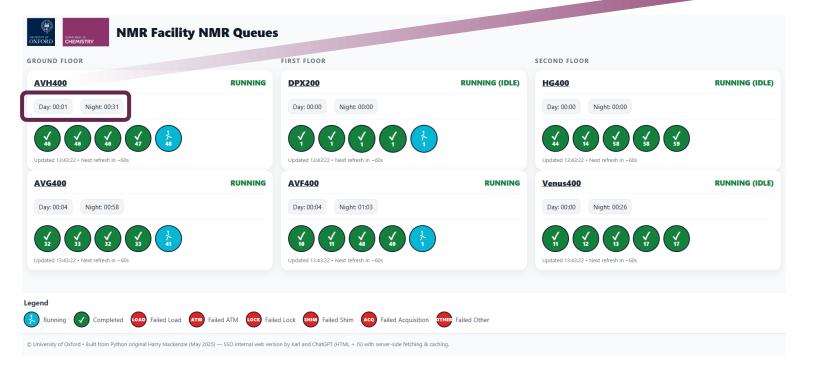




### Instrument Queues and Status

Open Access instrument queues(beta):

https://intranet.chem.ox.ac.uk/nmrq/





IconNMR Automation Run Status Day Experiments: 00:08 Busy Until: Tue 14:10 Night Experiments: 00:58

	STATUS			Expression			Trees
	Completed			N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)		00:01:32	
	Completed		1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)		00:01:32	
	Completed		1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)		00:01:32	
	Completed		1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)	djdgrp		
4	Completed	KC-811A	2	N CRL_FLUORINE 19F (1H coupled, 0 to 250 ppm, 16 scans)	djdgrp	00:00:48	Instru
	Completed		3	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)		00:01:32	
5	Completed	KC-\$11B	4	N CRL_FLUORINE 19F (1H coupled, 0 to 250 ppm, 16 scans)	djdgrp	00:00:48	Instru
6	Completed	CM-06-15RAC	1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)		00:01:32	
	Completed		1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)			
	Completed		1	n CRL_NOESY 1H-1H NOESY		00:24:00	
9	Completed		1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)	eaagrp		
11	Completed	GY-812 R2T3-12	1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)	vggrp	00:01:32	Instru
11	Completed	GY-812 R2T3-12	2	N CRL_FLUORINE 19F (1H coupled, 0 to 250 ppm, 16 scans)	vggrp	00:00:48	Instru
12	Completed	GY-812 R1T1-R2T2	1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)	vggrp	00:01:32	Instrus
12	Completed	GY-812 R1T1-R2T2	2	N CRL_FLUORINE 19F (1H coupled, 0 to 250 ppm, 16 scans)	vggrp	00:00:48	Instru
13	Completed	JDH-014-090-2-A-CDCL3 Sep29-2025-13-	1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)	ajrgrp	00:01:32	Instru
13	Completed	JDH-014-090-2-A-CDCL3 Sep29-2025-13-	2	N CRL_FLUORINE 19F (1H coupled, 0 to 250 ppm, 16 scans)	ajrgrp	00:00:48	Instru
3	Completed	JDH-014-090-2-A-CDCL3 Sep29-2025-13-	3	N CRL_FLUORINE_HDEC 19F (1H decoupled, 0 to 250 ppm, 16 scans)	ajrgrp	00:00:48	Instru
4	Completed	DWen363-363-V9	1	N CRL PROTON 1H (-4 to 16 ppm, 16 scans)	cjagrp	00:01:32	Instru
14	Completed	DWen363-363-V9	2	n CRL_COSY 1H-1H COSY	cjagrp	00:05:07	Instru
15	Completed	ALF138_dest2	1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)	eaagrp	00:01:32	Instru
23	Completed	KC-811Bafter	1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)	djdgrp	00:01:32	Instru
23	Completed	KC-811Bafter	2	N CRL_FLUORINE 19F (1H coupled, 0 to 250 ppm, 16 scans)	djdgrp	00:00:48	Instru
24	Completed	HA15435 400G	2	N CRL PROTON 1H (-4 to 16 ppm, 16 scans)	jwbgrp	00:01:32	Instru
25	Completed	KC-811SM	1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)	djdgrp	00:01:32	Instrus
25	Completed	KC-811SM	2	N CRL FLUORINE 19F (1H coupled, 0 to 250 ppm, 16 scans)	didgrp	00:00:48	Instru
16	Completed	Sep30-2025-26-LG-160-2	1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)	hlagrp	00:01:32	Instru
27	Completed	JDH-014-096-1-A-CDCL3 Sep30-2025-27-	1	N CRL PROTON 1H (-4 to 16 ppm, 16 scans)	airgrp	00:01:32	Instru
	Completed		1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)	vggrp	00:01:32	
28	Completed	AB-e253 epis 1-1 TEAF-U rt	2	N CRL FLUORINE 19F (1H coupled, 0 to 250 ppm, 16 scans)	vggrp	00:00:48	Instru
	Completed		1	N CRL PROTON 1H (-4 to 16 ppm, 16 scans)		00:01:32	
	Completed		2	N CRL FLUORINE 19F (1H coupled, 0 to 250 ppm, 16 scans)		00:00:48	
80	Error	KC-811B-mid	3	N CRL PROTON 1H (-4 to 16 ppm, 16 scans)		00:01:32	
30	Error	KC-811B-mid	4	N CRL. FLUORINE 19F (1H coupled, 0 to 250 ppm, 16 scans)			
	Completed		1	N CRL. PROTON 1H (-4 to 16 ppm, 16 scans)	ajrgrp	00:01:32	
	Submitted	Sep30-2025-31-JP82-F14	2	n CRL CARBON 13C (256 scans)	ajrgrp	00:14:33	
	Completed		1	N CRL_PROTON 1H (-4 to 16 ppm, 16 scans)	ajrgrp	00:01:32	
	Completed		2	n CRL CARBON 13C (256 scans)	ajrgrp	00:14:33	
	Completed		1	N CRL. PROTON 1H (-4 to 16 ppm, 16 scans)	ajrgrp	00:01:32	
	Submitted	Sep30-2025-33-JP82	2	n CRL CARBON 13C (256 scans)	ajrgrp	00:14:33	
	Completed		1	N CRL PROTON 1H (-4 to 16 ppm, 16 scans)		00:01:32	

	e Trees	Name :		of aremounty	Usen	Leade	Ame	Sees.	Loca	Senor	Aco F	Page Time	Kerman
42	13:52:26	Sep30-2025-42-MM-033	3	CRL_PHOSPHORUS_HDEC	ajrgrp							Instrument AVG400 Chemist Name Pinqi Wang Group AJ Project Account Code DMS000	R
42	13:48:38	Sep30-2025-42-MM6-033	1	CRL_PROTON	ajrgrp	-						Instrument AVG400 Chemist Name Pinqi Wang Group All Project Account Code DMS000	R
41	13:46:48	Sep30-2025-41-MM-028	3	CRL_PHOSPHORUS_HDEC	ajrgrp							Instrument AVG400 Chemist Name Pinqi Wang Group AJ Project Account Code DM8000	set to 0.0
41	13:41:55	Sep30-2025-41-MM-028	1	CRL_PROTON	sjrgrp	-	-		-	-		Instrument AVG400 Chemist Name Pinqi Wang Group AJ Project Account Code DMS000	
33	13:37:08	Sep30-2025-33-JP82	1	CRL_PROTON	NED							Instrument AVG400 Chemist Name Pinqi Wang Group AJ Project Account Code DMS000	R
32	13:19:33	Sep30-2025-32-JP82-F13	2	CRL_CARBON	ajrgrp	-	-			-	-	Instrument AVG400 Chemist Name Pinqi Wang Group AJ Project Account Code DMS000	R
33	13:14:45	Sep30-2025-33-LG-160-2-reox	1	CRL_PROTON	hlagrp			-		-		Instrument AVG400 Chemist Name Leonard Goh Group HLA Project Account Code DMR01590	
32	13:10:34	Sep30-2025-32-JP82-F13	1	CRL_PROTON	ajrgrp					-		Instrument AVG400 Chemist Name Pinqi Wang Group AJ Project Account Code DMS000	R
31	13:06:06	Sep30-2025-31-JP82-F14	1	CRL_PROTON	sjupp	-	4				•	Instrument AVG400 Chemist Name Pinqi Wang Group AJ Project Account Code DMS000	R
46	12:54:03	JR0277-THF-2h	2	CRL_PROTON_QUANT	eaagrp	-	100		-			Instrument AVG400 Chemist Name Jolia Ragus Group EAA Project Account Code dmt00240	
45	12:46:55	JR0277-PhMe-2h	1	CRL_PROTON_QUANT	eaagrp				Г	-	-	Instrument AVG400 Chemist Name Julia Ragus Group EAA Project Account Code day100240	
1	12:26:19	Sep30-2025-SB74 RET	1	CRL_PROTON	hlagrp	-	•			-		Instrument AVG400 Chemist Name Sara Borghi Group HLA Project Account Code dan/01590	
25	12:23:16	KC-8118M	2	CRL_FLUORINE	djdgrp	-						Instrument AVG400 Chemist Name Kathenine Clarke Group DJD Project Account Code other	sref: NUCLEUS '19F' not defined set to 0.0
25	12:19:05	KC-811SM	1	CRL_PROTON	djdgrp							Instrument AVG-00 Chemist Name Katherine Clarke Group D/ID Project Account Code other	
24	12:14:45	HA15435 400G	2	CRL_PROTON	jwbgsp	-	-		-	-	-	Instrument AVG400 Chemist Name Hon Lam Au Group JWB Project Account Code other Repeat-HA15435 P2 iso 400G	
40	12:08:50	Sep30-2025-40-ERC_P1FB1_chk	1	CRL_PROTON	hlagrp	-	-			-	-	Instrument AVG400 Chemist Name Edward Champness Group HLA Project Account Code DHT00110	
39	12:03:21	NC176_SMQ_actual	1	CRL_PROTON	18819		(2)					Instrument AVG400 Chemist Name Nicolas Charalambou Group VG Project Account Code dano 1860	
	11 50 33	Monay may		one promot								Instrument AVG400 Chemist Name Nicolas Charalambou	





### Organic Chemistry and Chemical Biology

10 NMR spectrometers available:

1 x 200 MHz: Open Access <sup>1</sup>H only

3 x 400 MHz: **Open Access** <sup>1</sup>H, <sup>13</sup>C, <sup>19</sup>F, <sup>31</sup>P, routine 2Ds

1 x 400 MHz: Hands on

2 x 500 MHz: Hands on and Service

1 x 600 MHz: Service

1 x 600 MHz: Hands on, Service, Research Projects

1 x 700 MHz: Hands on, Service, Research Projects





### Organic Chemistry and Chemical Biology

#### **Open Access**

#### **Ground Floor**



**AVG400** 



**AVH400** 

#### **First Floor**



AVF400



**DPX200** 





### High-Field Facilities



Basement high-field NMR lab

400 and 500 MHz instruments available for 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 of instrument time: <a href="https://booking.chem.ox.ac.uk/">https://booking.chem.ox.ac.uk/</a>





#### NMR Submission Service

#### Availability of experiments

Other nuclei: <sup>2</sup>H, <sup>7</sup>Li, <sup>11</sup>B, <sup>27</sup>Al, <sup>119</sup>Sn etc.

2D experiments: TOCSY, H2BC, ROESY, <sup>1</sup>H-<sup>15</sup>N HSQC/HMBC

More specialised experiments: DOSY, variable temperature, pure

shift, selective and more

#### 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







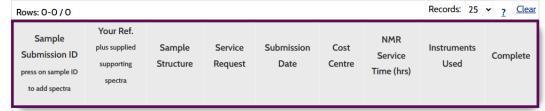
### NMR Submission Service (New System)

NMR Sample Submission Service v24 Nov 2023

Welcome Edward Tomlinson (NMR)

To submit a new sample, fill in this online form

List of your previous sample submissions and their status:





iample for: Mr Edward Tomlinson ( edward.tomli	nson <b>@</b> chem.ox.ac.uk )	
Date of Submission: 11/09/2025		
Group: NMR Role: SRF Staff	Phone: <b>75659</b>	Lab/Office: CRL -00.086
our Sample Ref Code/Number:	Project Cost Centre Code:	
	Other - add into description	n <b>~</b>
xperiments:		
] 1H   13C   19F   31P   COSY   HSQC	HMBC   NOESY   Other (describe	e)
ndicate Hazards:	Mass Supplied (mg):	Solvent
none unknown toxic carcinogen		Chloroform-d 🔻
iample Location: O Rack O Fridge O Request		
Describe the experiments that you would like the	NMR service to run for you:	
		_
Upload a picture of your exped	cted sample chemical structure, drag	and drop to this area works.
Choose F	File No file chosen	





### **Inorganic Chemistry**

5 NMR spectrometers available:

1 x 400 MHz: Open Access multinuclear

1 x 400 MHz: Open Access multinuclear

1 x 500 MHz: Hands on & Service multinuclear and VT work

1 x 400 MHz: Service Solid State HXY

1 x 400 MHz: Service Solid State HFX, microimaging & diffusion

Access to 600 MHz: Service multinuclear





### **Inorganic Chemistry**

#### **Open Access**

#### **Second Floor**



**AVG400** 



**AVH400** 

#### **Hands-on**

#### **Basement**



**AVD500** 





#### Solid state NMR

- Service provided by Dr Nick Rees
- Stable samples provided as a powdered solid (c.a. 200mg)
- Unstable samples can be packed in glove box
- Consult Nick Rees <u>before</u> 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.





### 2 x 400MHz Wide Bore 3 channel systems



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



HFX400

- 3.2mm HFX Triple Magic Angle Spinning Probes (<sup>1</sup>H, <sup>19</sup>F, <sup>31</sup>P to<sup>15</sup>N)
- 30mm Micro-imaging probe
- Diffusion probe (-150 to 200C)
- Variable temperature capable (-150 to 150C)

**HFX400** 

## university of OXFORD

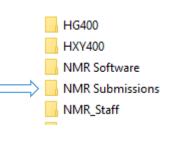
#### Solid State NMR Service

Sample Submission

Form: Word document

List experiments
And specify SSNMR

If the sample needs to be packed in glove box put down as request





Solids Submission

CHEMISTRY RESEARCH LABORATORY
NMR SERVICE

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:¹

Submission Number.² 70957

Project Code/ Charge Account:³

DHT00110 DHSA.08

Submission Date: 06/07/23

Nuclei of interest:

Structure:

Toxicity: ² unknown

Solution Depths.³

Max 4.5 cm

Min 4.0 cm

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

Nature of problem.⁴ expected 11B shift around +10-+60 ppm, expected 31P at approx. -10 ppm

Mass supplied:¹

Solvent.⁴

Referencing: '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>4</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 experiments) 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.





### **Future Training Courses**

Use of the Open-Access NMR Spectrometers & Service

Running this week and next: meet in CRL reception

Compulsory sessions: you must attend before using instruments or the NMR submission service. Thurs  $2^{nd}$  (pm), Mon  $6^{th}$  (pm), Tues  $7^{th}$  (am & pm).

#### MestreNova 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 Canvas at: <a href="https://canvas.ox.ac.uk/courses/54457">https://canvas.ox.ac.uk/courses/54457</a>





### NMR Training for New Researchers

Organic/Chemical Biology Sections	Inorganic Sections
Register as a new user:  New Organic/Chembio NMR User	Register as a new user:  New Inorganic NMR User
Book a training session:  Organic NMR Training	Book a training session:  Inorganic NMR Training

#### **Meet for training in CRL reception**

To arrange training external users should email: <a href="mailto:nmrstaff@maillist.chem.ox.ac.uk">nmrstaff@maillist.chem.ox.ac.uk</a>

All the above links can be found in these slides online at: <a href="https://nmr.chem.ox.ac.uk/">https://nmr.chem.ox.ac.uk/</a>





### QR for Training Sign-up Sheets

