

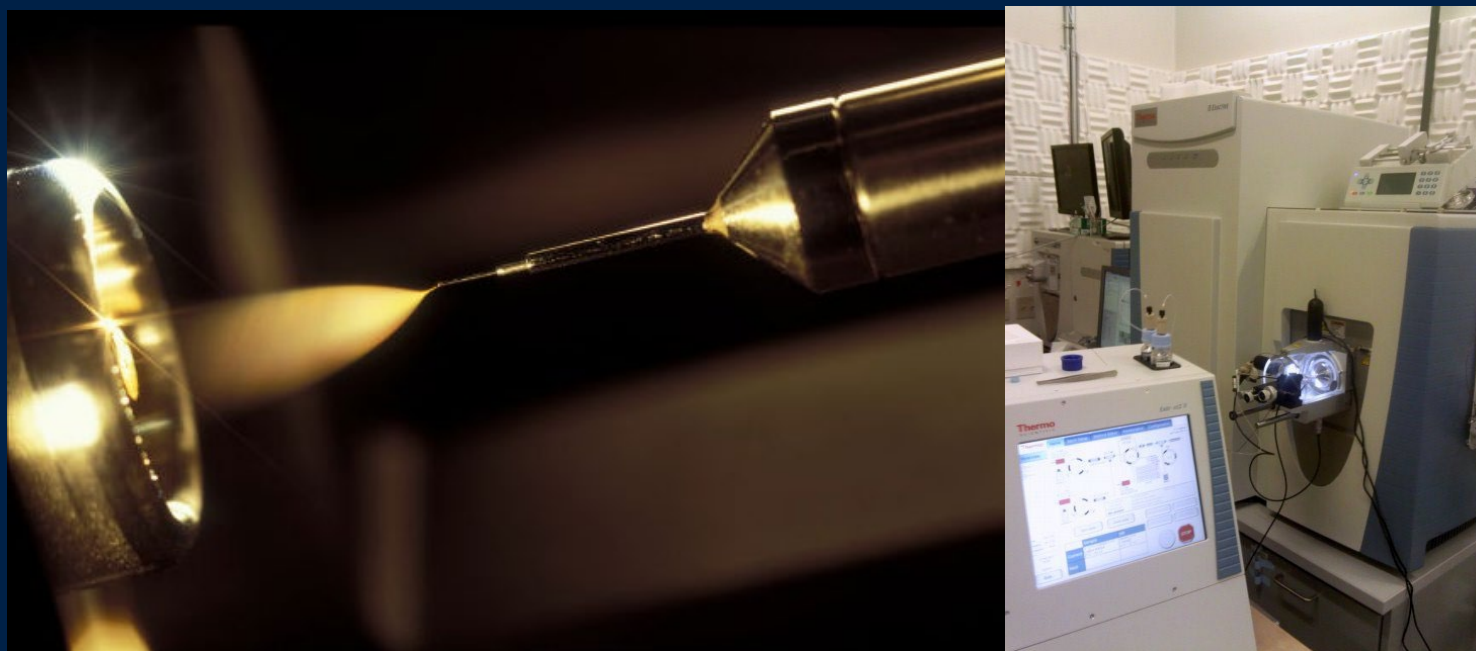
SCIENTIFIC INDUCTION SESSIONS

9.00 - 9.30	<p>Introduction to Mass Spectrometry facilities</p> <p>The Mass Spectrometry induction session will introduce researchers to the mass spectrometry facilities available to support research the Chemistry Department. It will explain how these can be accessed including Open Access Mass spectrometer systems, training to use LC-MS and GC-MS and how the sample submission services are arranged and run by the mass spec staff. <i>It is essential that anyone planning to use the Mass Spectrometry facilities attend this induction session.</i></p>	<p>James McCullagh & John Walsby-Tickle</p>
9.30 - 10.15	<p>Introduction to NMR Spectroscopy facilities</p> <p>The NMR induction session will introduce the NMR staff, instruments, and facilities available to support research in organic chemistry, chemical biology and inorganic chemistry. It will explain how these can be accessed, the training that is required to use the instruments, and will introduce the NMR Submission Service provided by the NMR staff. <i>It is essential that anyone wishing to make use of the NMR facilities attend this induction session</i></p>	<p>Nick Rees</p>
10.15 – 10.30	<p>Break</p>	
10.30 – 10.50	<p>Introduction to X-Ray Crystallography</p> <p>A short introduction to the single crystal X-ray crystallography service suitable for all Part II and new DPhil students. This session is relevant to chemists doing synthetic work in all sections of the Department.</p>	<p>Richard Cooper</p>
10.50 – 11.00	<p>Introduction to Powder X-Ray Diffraction and Magnetometry</p> <p>A brief introduction to the Inorganic Materials Characterisation facility which encompasses PXRD and SQUID magnetometry. An overview of the equipment and an explanation of the induction process will be given, relevant to all new starters undertaking synthetic work.</p>	<p>Simon Cassidy</p>
11.00 – 11.20	<p>Introduction to ESR Spectroscopy facilities</p> <p>The ESR induction session will introduce the ESR staff, instruments, and facilities available to support research in chemical biology and inorganic, organic, and physical chemistry. It will explain how the instruments can be accessed and the training that is required to use them.</p>	<p>Will Myers</p>

DEPARTMENT OF CHEMISTRY
MASS SPECTROMETRY RESEARCH FACILITY



INTRODUCTION TO THE MASS SPECTROMETRY RESEARCH FACILITY



Prof James McCullagh, Director of the Mass Spectrometry Research Facility

Dr John Walsby-Tickle, Mass Spectrometry Services Manager

MASS SPECTROMETRY RESEARCH FACILITY

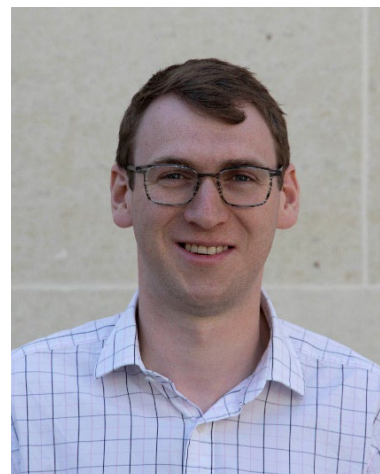
Mass Spectrometry Research Facility and staff support Research in the CRL



Ms Elisabete Pires
Research Associate in Mass Spectrometry



Dr Victor Mikhailov
Research Associate in Mass Spectrometry



Dr John Walsby-Tickle
MS Services Manager



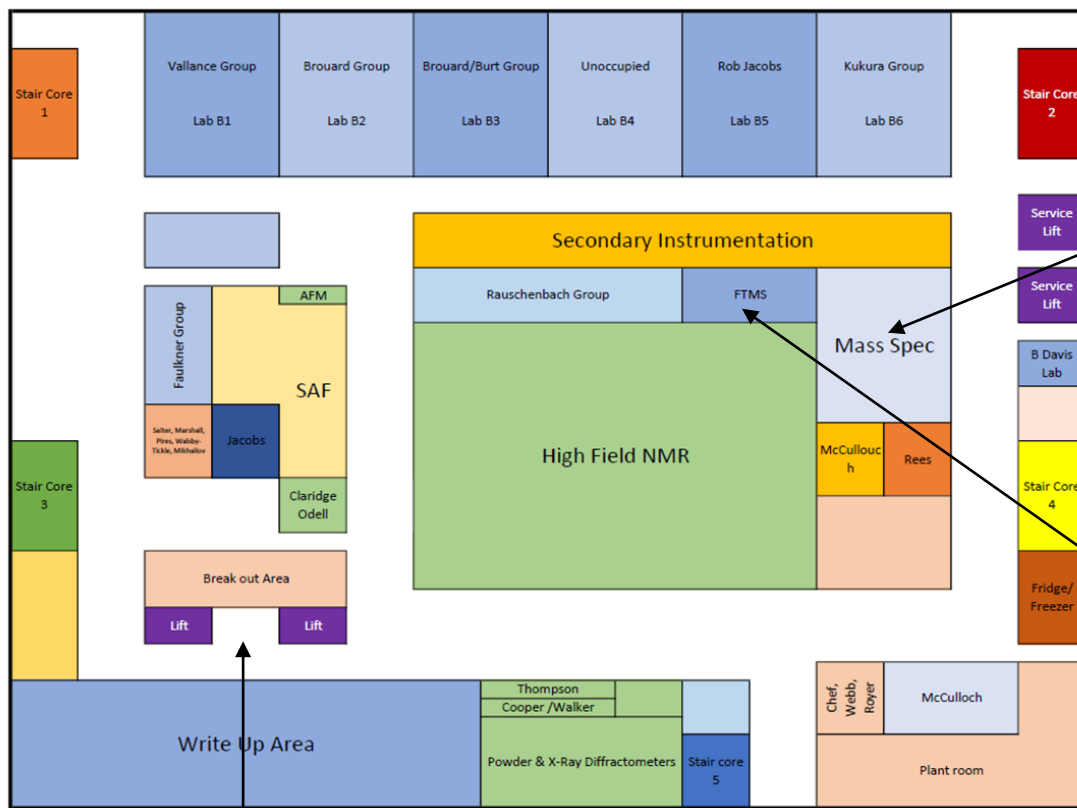
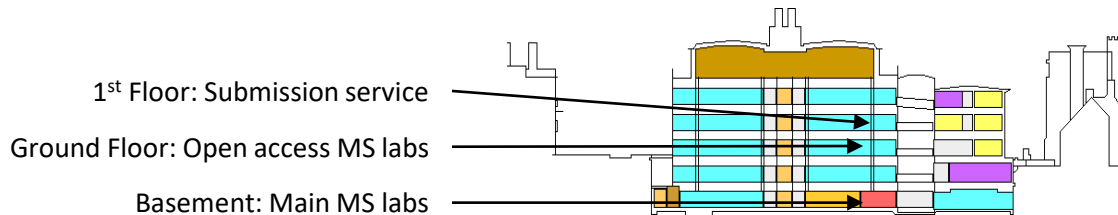
Prof James McCullagh
Director of the Mass Spectrometry Facility

20+ MASS SPECTROMETERS AVAILABLE IN CRL

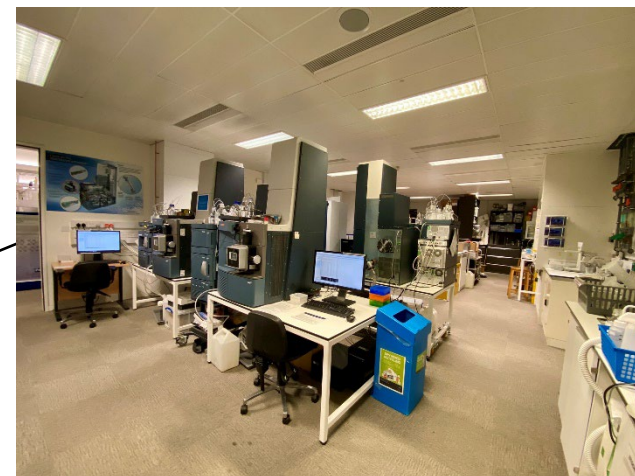


Discuss with a member of MS staff what might be the most appropriate mass spectrometry approach for your experimental aims.

LOCATION OF THE MASS SPECTROMETRY MAIN LAB IN THE CRL



Scenic lifts

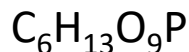


MASS SPECTROMETRY

Mass spectrometers measure the mass to charge ratio of ions in the gas phase

Elemental composition and Identification

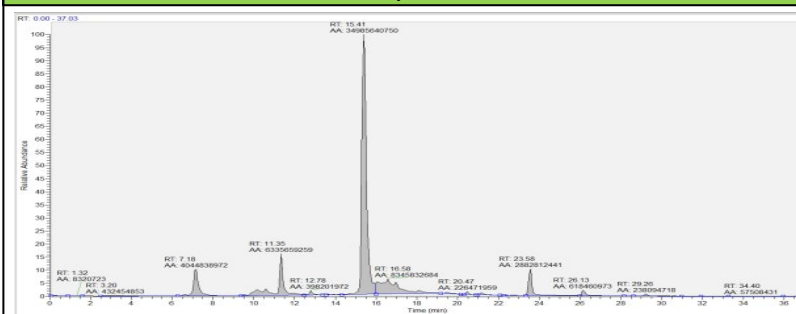
260.0297185



Glucose-1-phosphate

Separation & Quantification

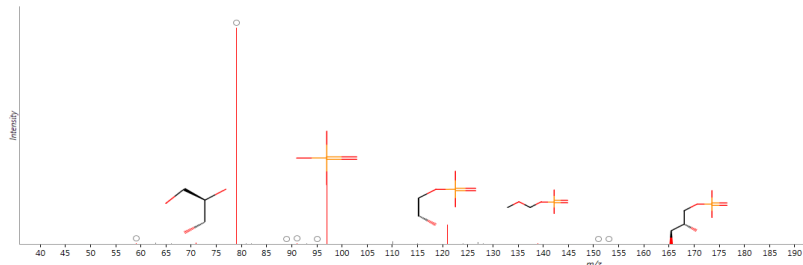
Relative and absolute
LC-MS, GC-MS



Structural analysis

Fragmentation studies using MS

Compound 11.35_241.0109m/z (Glucose-6-Phosphate)



'Omics', structural, synthetic and systems biology



4 DIFFERENT WAYS TO ACCESS MASS SPECTROMETRY

1) Open access systems (x5)

BioAccord
1

BioAccord
2

Open
access - A

Open
access - B

Open
access - C

CRL, ground floor mass spectrometry lab

2) Mass spectrometry services

Accurate
Mass Service

Alternative
ionisation
Service

Biological
molecules
Service

3) trained user MS systems x10

LC-MS

LC-MS

LC-MS

LC-MS

LC-MS

GC-MS

MALDI

Glove-Box
- MS

Triple quad

GC-MS

4) Collaborative MS experiments

Proteomics
Bottom up and top down

Metabolomics
Targeted and untargeted

Ion-mobility MS

Native state protein MS

High-throughput screening
by MS

MALDI imaging

Quantitation

Thermal desorption GC-MS

OPEN ACCESS MS SYSTEMS – GROUND FLOOR (X5)



Victor Mikhailov

Research Associate in Mass Spectrometry

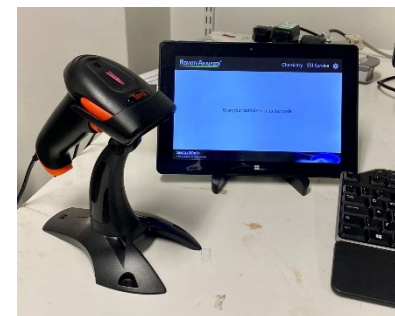
Runs open access systems and small molecule services

Ground Floor



SpectralWorks

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



Target Confirmation Report

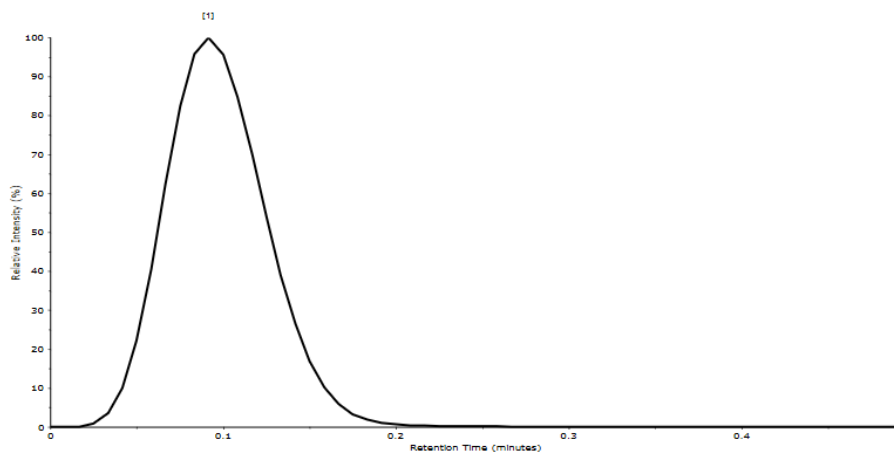
Sample ID: LeuEnk
 Group: Mass Spec
 Acquisition Date: 03/08/2022 08:44:30
 Experiment: BioAccord 2 - Loop Injection MS (+ ion)
 Filename: Mass_Spec_JWT_LeuEnk_1575_C28H37N5O7_(+H)+.pdf

Submitter: John Walsby-Tickle
 Project: BioAccord 2 - Accurate Mass Confirmation
 Instrument: BioAccord 2

Adduct: +H
 Proposed Formula: $C_{28}H_{37}N_5O_7$

Additional Comment:

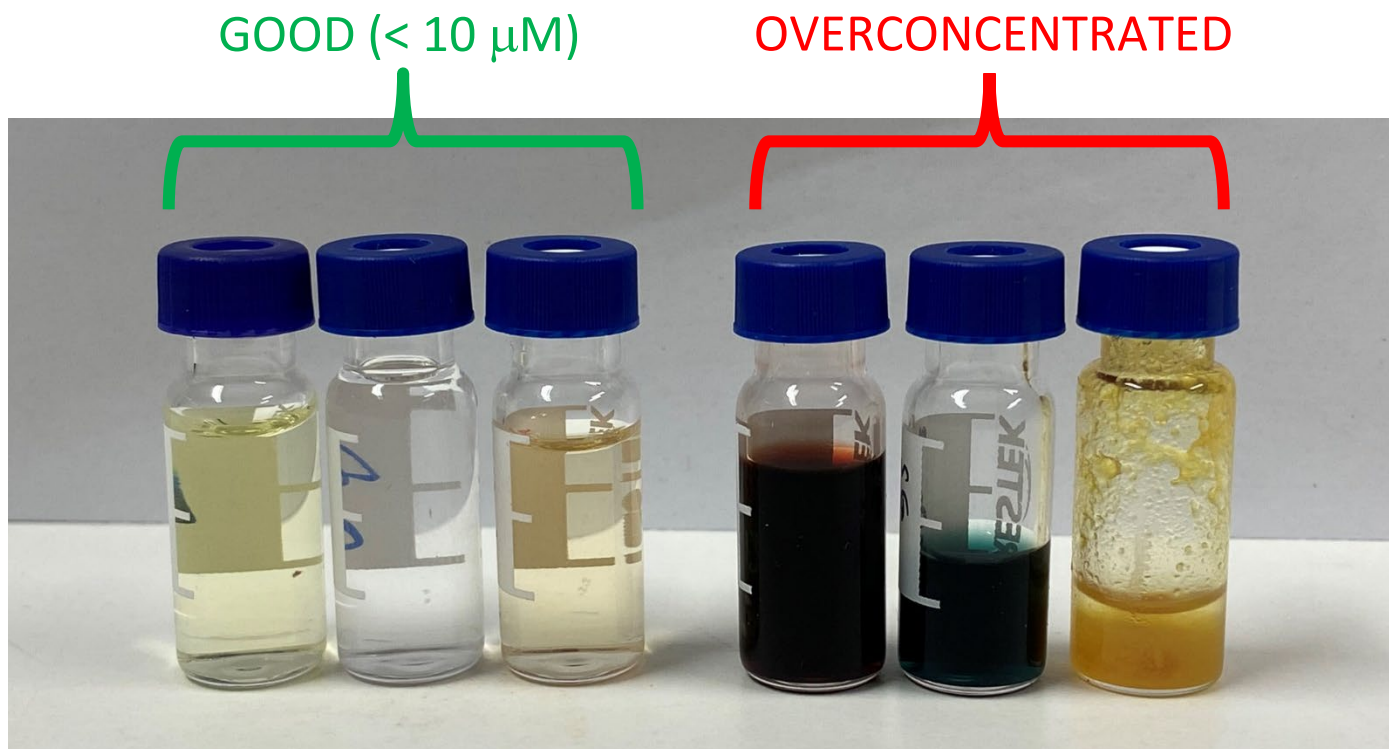
EIC $m/z=556.2766 \pm 0.0500$, Target Mass and BasePeak Peaks Align, 1 Peaks Detected, NL 6.868E06



REMOTEANALYZER

<input type="checkbox"/>	↓ ID	Sample Refere...	≡ Status	Result ≡
<input type="checkbox"/>	2921	OMP040-C	■■■■■■■■	Target m/z 334.1562 not found, Target ...
<input type="checkbox"/>	2919	JTW-3-045-Ax	■■■■■■■■	C96H119N12O2, 3.3 Error (ppm)
<input type="checkbox"/>	2918	EF001A	■■■■■■■■	C26H51N4O6, 3.5 Error (ppm), C26H50...
<input type="checkbox"/>	2917	SM-8-41	■■■■■■■■	C24H39BF3O3Si, 1.7 Error (ppm), C24...
<input type="checkbox"/>	2916	SM-8-35	■■■■■■■■	C14H27OSi, 1.8 Error (ppm), Target $m/...$
<input type="checkbox"/>	2915	dmjc734C	■■■■■■■■	C28H37O5, 2.1 Error (ppm) [S], C28H36...
<input type="checkbox"/>	2914	dmjc734B	■■■■■■■■	C26H41O5, 0.8 Error (ppm), C26H40O5...

SAMPLE PREPARATION FOR SMALL MOLECULE OPEN ACCESS



BioAccords: <https://masspec.chem.ox.ac.uk/files/spp-ba-targetconfirmation>

ESI Service: <https://masspec.chem.ox.ac.uk/files/esi-servicespppdf>

4 DIFFERENT WAYS TO ACCESS MASS SPECTROMETRY

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Biological
molecules
Service

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LC-MS

LC-MS

LC-MS

LC-MS

LC-MS

GC-MS

MALDI

Glove-Box
- MS

Triple quad

GC-MS

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Ion-mobility MS

Native state protein MS

High-throughput screening
by MS

MALDI imaging

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Thermal desorption GC-MS

SMALL MOLECULE CHARACTERISATION SERVICE

SMALL MOLECULE SERVICES (ORGANIC AND INORGANIC):

1. **ESI Accurate Mass Service** (uses electrospray ionisation)
2. **Alternative Ionisation Service** (uses multiple ionisation types)

First Floor



Dr John Walsby-Tickle
MS Services Manager



Dr Victor Mikhailov
Research Associate

NB: Samples must be analysed on an open access system before submitting to the Small Molecule Services

LARGE MOLECULE CHARACTERISATION

BIOLOGICAL MASS SPECTROMETRY:

1. Proteomics Services
2. Native state protein MS
3. Oligonucleotide analysis
4. MALDI peptide and protein MS



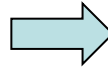
Ms Elisabete Pires
Research Associate in biological
mass spectrometry

SAMPLE SUBMISSION BIOLOGICAL SAMPLES

If you have not used biological service before you must first discuss your research requirements with Elisabete Pires.



Elisabete.pires@chem.ox.ac.uk



MASS SPECTROMETRY RESEARCH FACILITY

Department of Chemistry
University of Oxford
Rutherford Road, Oxford
Telephone: +44 (0)1865 275
pires.mrc@chem.ox.ac.uk

MS Facility Sample Submission Form:
Proteomics samples

Please complete 1 form per sample
The form can be found at: <http://www.chem.ox.ac.uk/electrometry/mass-spec/index.html>

Name/Company name: _____ Telephone: _____
Group: _____ email: _____
Project Code/Charge Account: _____ Date: _____

Sample Information

Sample Type: Digest (in sol) Digest (in gel) Cell lysate Gel band Purified prot

Sample ID: (5 characters or less and as appears on vial) _____
Enzyme used for digestion: _____
Sample volume supplied: _____
Amount of protein digested: _____
Protein name: _____
Sample/buffer solvent: _____
Organism protein in form: _____

Analysis Type **Analysis Required** **Please send/bring amino acid sequence for data analysis**

Untargeted MS/MS (default method)
 Targeted MS/MS (please provide precursor (target) mass)

Other information or special instructions: _____

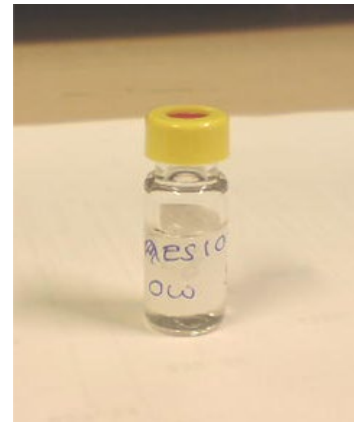
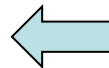
MS Research Facility: Proteomics Service, 1st Floor, 10th Avenue, Oxford, UK

*Note: a Project code or Charge account is now compulsory as of 5th Aug 2015. If you do not know which project code to use please discuss with your supervisor or the CEM, (Oxford team). Samples WILL NOT be analysed without a valid code being entered.

Complete PDF proteomics sample submission form



It can take up to 4 weeks to analyse samples. You should contact Elisabete to check your analysis has completed successfully.



Bring sample and completed form down to basement MS lab and submit to Elisabete in person. There is a freezer for storage of samples.

Proteomics protocols and submission form can be found on the website:

<https://masspec.chem.ox.ac.uk/proteomics-service>

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GC-MS

MALDI

Glove-Box
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Triple quad

GC-MS

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Bottom up and top down

Metabolomics
Targeted and untargeted

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Native state protein MS

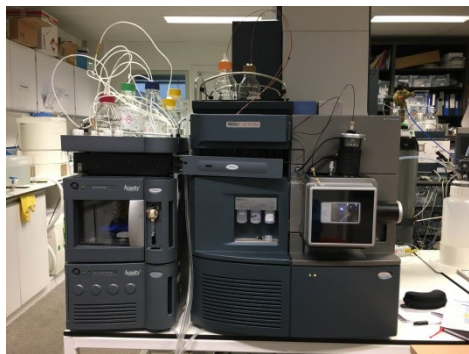
High-throughput screening
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TRAINED-USER MASS SPECTROMETRY (4X LC-MS SYSTEMS)



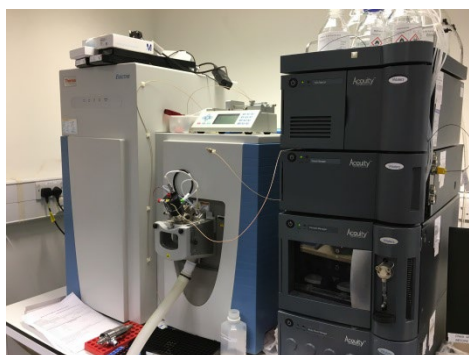
Waters UPLC – Xevo G2-XS QTOF



Waters UPLC – Xevo G2-S QTOF

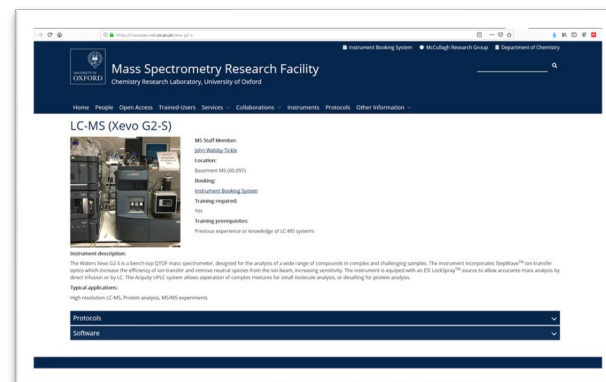


Waters UPLC – Xevo G2 QTOF (Oligo)



Waters UPLC – Exactive Orbitrap

- Complex mixture analysis
- Amino acid analysis
- Natural product identification
- Metabolomics
- Proteomics
- Oligonucleotide MS



Protocols and procedures MS Facility website and as part of training

MALDI (TOF-TOF) & GC-MS MASS SPECTROMETRY

Matrix-Assisted Laser-Desorption Ionisation (MALDI): A laser based ionisation technique useful for analysis of biological and synthetic polymers



Bruker Autoflex Speed TOF-TOF

- Peptides/proteins
- Oligonucleotides
- Polymers.
- Small molecules above ~450 Da.

GC-MS: Useful for high resolution, high sensitivity separation of compounds in the gas-phase with identification based on IE mass spectral libraries.



Agilent GC-MS (quad)

- Complex mixture analysis
- volatiles
- SPME
- Headspace analysis
- Metabolomics

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LC-MS

LC-MS

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Triple quad

GC-MS

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Native state protein MS

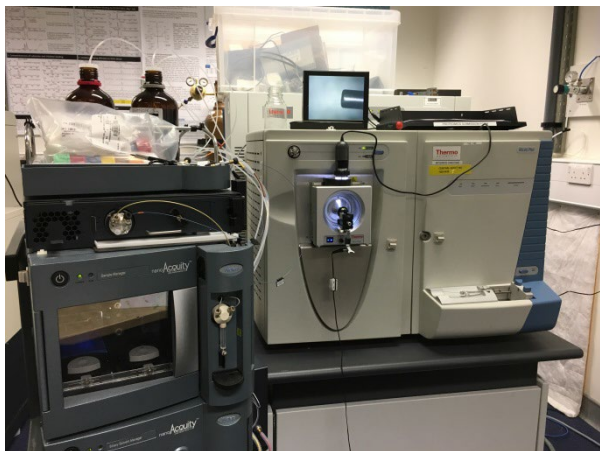
High-throughput screening
by MS

MALDI imaging

Quantitation

Thermal desorption GC-MS

COLLABORATIVE MASS SPECTROMETRY EXPERIMENTS



Proteomics

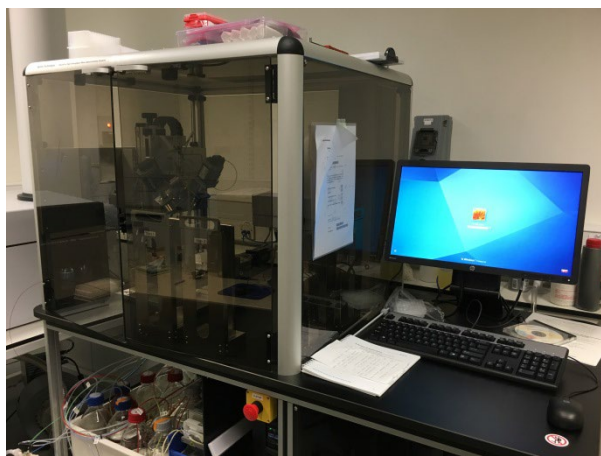


Inert atmosphere MS

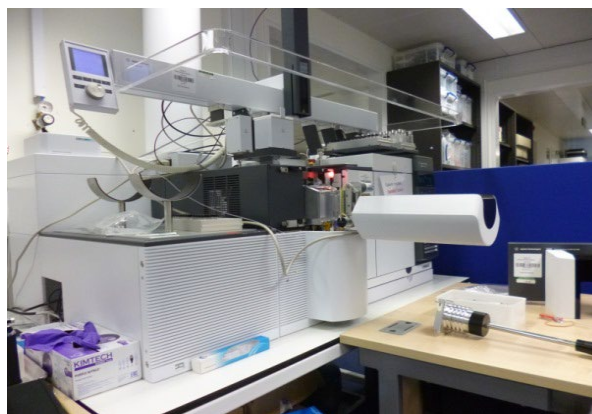


SYNAPT ion mobility MS

Collaborative Experiments



Small molecule screening
(Rapidfire)



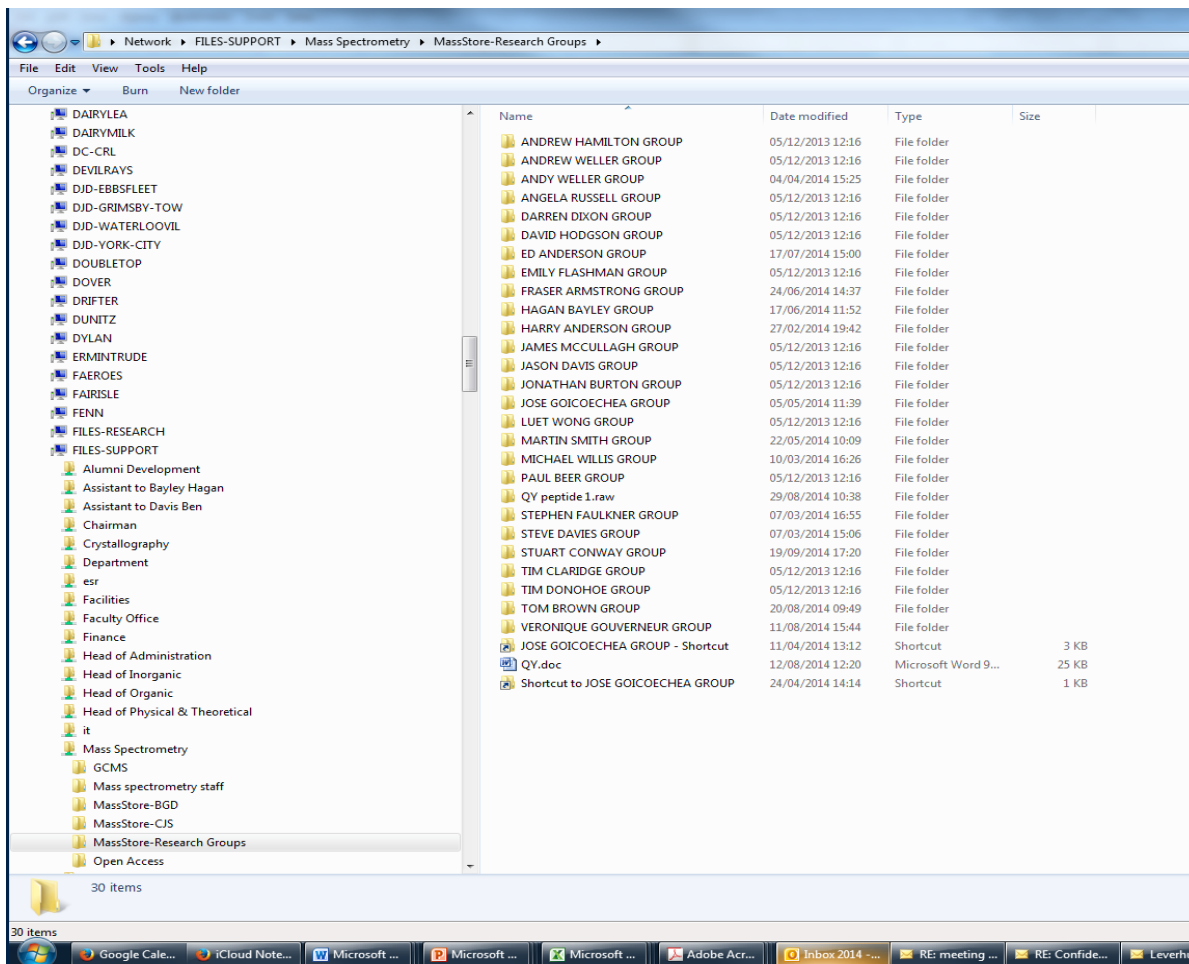
Solids Probe MS



Metabolomics

DATA STORAGE: MASS-STORE ('Q: DRIVE')

High resolution MS experiments, particularly LC-MS and GC-MS create large data files.



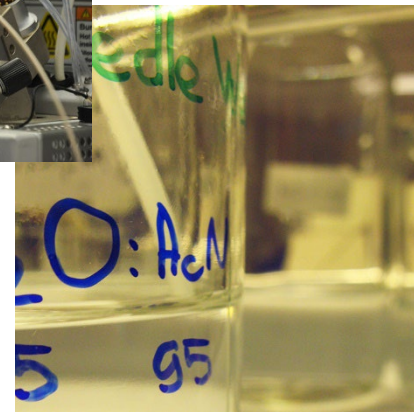
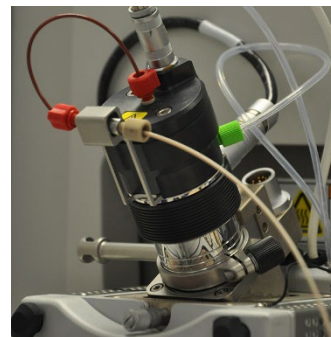
The MS Facility has a networked server with over 150 TB of storage space for storing your data from all types of MS system.

Each research group has a folder which you can create your own personal folder for MS data.

This has RAID redundancy and is mirrored on a separate drive system in two buildings.

PROTOCOLS AND SAFETY IN THE MS LABS

- No lab coats to be worn in any of the MS labs.
- Never remove or modify any part of the mass spectrometer, sample inlet system or software.
- If something is not working properly or appears to be broken please report it to a member of the MS Facility staff.
- Please read safety information on the mass spectrometry website and all information provided on laminated sheets around the instruments.
- Follow the booking rules outlined on the facility website



MASS SPECTROMETRY RESEARCH FACILITY WEBSITE



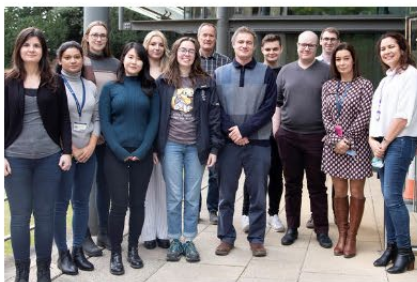
Mass Spectrometry Research Facility

Chemistry Research Laboratory, University of Oxford

Status Page Booking System McCullagh Research Group Department of Chemistry Login



Home People Open Access Trained-Users Services Collaborations Instruments Protocols Other Information



People



Open Access



Trained-Users



Services



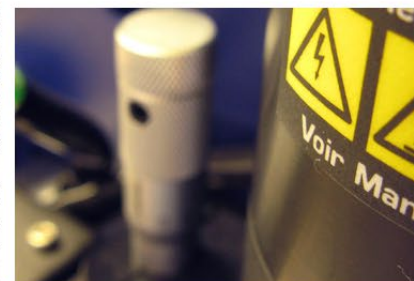
Collaborations



Instruments



Protocols



Other Information

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

TRAINING COURSES IN MASS SPECTROMETRY

MS open access systems

To start using the MS open access systems and submission services you will need to complete some online training followed by short in-person induction session. You must complete the training before using open access MS instrumentation and services.

In-person inductions

Tue 3rd October - 1pm, 2pm, 3pm

Thu 5th October - 1pm, 2pm, 3pm

Fri 6th October - 9am, 10am, 11am

Other instrument training

See website for information on trained user instruments and how training is organised. If you require training please contact John Walsby-Tickle.

john.walsby-tickle@chem.ox.ac.uk



QR code takes you to the booking forms for the training sessions.

HERE TO HELP YOU WITH MASS SPECTROMETRY



Dr Victor Mikhailov
Research Associate

Tel: 75687, victor.mikhailov@chem.ox.ac.uk

Areas of focus

MALDI
Accurate Mass Analysis
Native State Proteins
Open Access
Probe MS
MS services



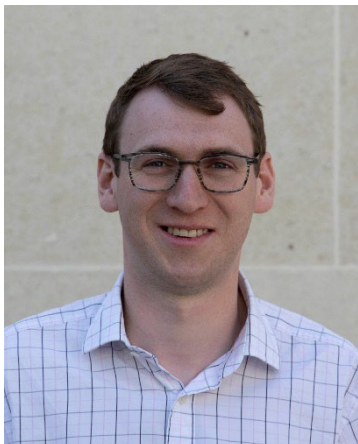
Ms Elisabete Pires

Research Associate (biological MS)

Tel: 75942, elisabete.pires@chem.ox.ac.uk

Areas of focus

Proteomics
Metabolomics
Biological MS
LC-MS
MALDI (biological)
Sample prep



Dr John Walsby-Tickle

Mass Spectrometry Services Manager

Tel: 85025, john.walsby-tickle@chem.ox.ac.uk

Areas of focus

Manages training & maintenance
Small molecule analysis
Metabolomics
Infrastructure
IT and software for MS



Prof James McCullagh

Director of the Research Facility

Tel: 75657, james.mccullagh@chem.ox.ac.uk

Areas of focus

LC-MS
Metabolomics
Isotope tracer experiments
Orbitrap MS
Sample preparation
Quantitation

QR FOR MS AND NMR TRAINING



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