**University of Glasgow**

**Guidelines for users of the Mass Spectrometry Facility (School of Chemistry)**

**General Information:**

The service is located within the Joseph Black Building, Laboratory B2-25.

The telephone numbers for the service are: External: 0141-330-8609 and Internal Ext: 8609.

Sample Submission Forms are obtained at: <http://www.gla.ac.uk/media/media_364016_en.docx>

**Instrumentation**

**JEOL MStation JMS-700**: This instrument is a high resolution magnetic sector mass spectrometer which has Electron Ionisation (EI), Chemical Ionisation (CI) and Field Desorption (FD) acquisition mode options and has the ability to produce mass measurements with typically < 5ppm accuracy, using internal calibration.

**Applications:**

**Electron Ionisation (EI)** - For volatile and semi-volatile molecules normally in the mass range 50 to 800 Da

**Chemical Ionisation (CI)** - For molecules requiring a softer ionization than EI, normally in the mass range 50 to 800 Da.

When performing accurate mass measurements using the above techniques an initial low resolution analysis is required, with the purpose of obtaining a spectrum with a molecular ion (or adduct of), prior to the accurate mass analysis.

**Bruker MicroTOFq:** This instrument has an Electrospray (ESI) ion source, coupled to a time-of-flight (TOF) analyzer and is capable of analysis using positive and negative ion modes.

**Applications:**

**Electrospray Ionisation (ESI)** – Soft Ionization technique used for the analysis of non-volatile and thermally labile compounds, producing spectra with very little fragmentation (occasionally only the molecular ion or adduct). Positive ionization relies on protonation and is suitable for compounds containing basic functional groups, typically producing (M+H)+ and (M+Na)+ ions. Negative ionization relies on deprotonation and therefore is suitable for compounds containing an acidic functional group, typically producing (M-H)- ions.

This instrument can produce accurate mass data with typically < 5ppm accuracy, over the full spectral range, offering a much quicker analysis time than EI or CI accurate mass analysis.

**Shimadzu LCMS – 2010EV**: Liquid Chromatograph Mass Spectrometer (LCMS) system with an ESI source that can operate in positive and negative ionisation modes. The LC system also incorporates a dual wavelength UV/VIS detector.

**Application**

The instrument can be used for the quantitative analysis of extracts containing a range of organic non-volatile and thermally fragile molecules. Applications include the analysis of food and environmental sample extracts for trace levels of pesticides, antibiotics, drugs and metabolites.

**Shimadzu GCMS**: This system is a Gas Chromatograph Mass Spectrometer (GCMS) that can operate in EI mode..

**Application**

The instrument can be used for the quantitative analysis of a wide range of volatile and semi-volatile mixtures. Applications include the analysis of sample extracts for trace levels of polycyclic aromatic hydrocarbons (PAHS) and pesticides.

**Solvents**

Electron Ionization (EI) / Chemical Ionization (CI)

Dichloromethane and Methanol will be the preferred solvents for samples submitted for EI and CI analysis. Solvents such as DMF and DMSO are best avoided, but can be used if there is no other alternative.

Electrospray (ESI)

Acetonitrile and Methanol will be the preferred solvent for samples submitted for ESI analysis however other solvents including acetone, THF, ethyl acetate and dichloromethane are also acceptable. Samples submitted for ESI analysis should not contain buffers or trifluoroacetic acid (TFA).

**Procedure for Sample Submission**

**Sample Submission Form:**

A sample submission form <http://www.gla.ac.uk/media/media_364016_en.docx> should be completed accurately for each sample, giving as much information as possible. Please DO NOT include more than one sample per submission form.

Please ensure that the Reference Number given on the sample vial is the same as the Reference Number entered into the form.

Check that information entered for Structure, Molecular Formula and Nominal Mass are correct – incorrect information can result in wasted analysis time.

It is essential that a suitable solvent be specified for the analysis type requested (see above for information on solvent choice).

If you are unsure of a suitable *ionization technique*, please check the “No Preference’ box and the service analyst will choose the most appropriate.

Please note that LCMS and GCMS options should only be checked after consultation with either Jim Tweedie or Gangi Reddy Ubbara. (These instruments can be used on an open access basis after appropriate training).

**Submission of Samples:**

Samples, accompanied with a completed submission form, should be placed in the Mass Spectrometry Submission Rack in Laboratory B2-25. If not labeled clearly, samples will be returned for re-submission.

Sample vials should be glass with screw caps (2ml vials are ideal). Please do **NOT** submit samples in NMR tubes, crimped autosampler vials or plastic containers.

It is preferred that samples are submitted neat and a choice of solvent given in the submission form.

Where submission of a sample in solution is unavoidable, please identify the solvent and give an approximation of the concentration of analyte and any other materials present.

If samples are unstable and require cold storage, a freezer is available – remember to indicate this on the submission form. Unstable samples will also be given priority and analysed without delay where possible.

**Reporting of Analysis Results**

Analysis results and samples will be placed in the Mass Spectrometry Submission Rack in Laboratory B2-25. In certain circumstances ESI analysis results can be reported by email (.pdf file), however useful comments regarding anomalies of the analysis and spectral interpretation will not be included. Results and samples not collected within three weeks will be discarded.