

# Research services

## Analytical facilities

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**Laboratory Accreditation** Within SCRI, the Gas Chromatography-Mass Spectrometry Laboratories, Stable Isotopes Facility and Lipid Analysis Unit of MRS Ltd, operate a formal Quality System certified to BS EN ISO 9001 by SGS Yarsely International Certification Services Ltd. The certification standard was upgraded from ISO 9002 to ISO 9001 in August 1999, and now includes the design and conduct of research within its scope. A generic Quality System operates in other parts of the Institute and this is summarised in the SCRI Quality Plan, a copy of which is included in the Institute's Corporate Plan. The measures required for implementation of the generic SCRI Quality System are described in a Code of Practice document, a copy of which is issued to all members of staff. The Code of Practice is also reproduced at the end of the latest version of the SCRI Laboratory Notebook. The generic system is based on the correct maintenance of work records in which specially designed hardback notebooks comprise the primary record, with other data recording systems, archival procedures etc. as secondary records. The preparation of written methods or protocols (Standard Operating Procedures) and the correct use of equipment and facilities are strongly encouraged. The plan ensures full compliance with all safety regulations, and demands high standards of laboratory hygiene. If required, the Quality System can be readily upgraded to the standard required for formal certification within any activity or area. An archive facility is located within Building S and this is used for long-term storage of data as hard copy and in electronic format. Archival of data on electronic media is based on the use of compact disc writers (CD-R format) installed in several personal computers. Data can be transferred over the network or from a portable high capacity data storage disc to the computer's hard disc, and then to CD. Each CD can hold up to 650 Mbytes of data. Two copies are made, one for the owner of the data and one for the archive.

**Stable Isotope Facility** Stable isotopes are widely used for the study of plant physiology, crop genetics, ecology and food webs. Valuable information comes

both from studying natural variation in stable isotope composition and from following the fate of added isotopic tracers. SCRI is equipped with a comprehensive range of modern instrumentation for stable isotope analysis of the biologically important light elements,  $^{13}\text{C}$ ,  $^{15}\text{N}$ ,  $^{18}\text{O}$  and  $^{34}\text{S}$ , in a wide range of solid, liquid and gas samples.

All the instrumentation is based on continuous-flow isotope-ratio-mass spectrometers that are fully automated and operated through computer data systems. Automation allows a high through-put of samples, essential for many biological experiments where large data sets are required. For solid samples, the Europa Scientific Tracermass and 20-20 mass spectrometers are interfaced to Roboprep CN and ANCA-NT SL combustion sample converters. A Roboprep G+ gas purification unit is used for gas analysis. Plant samples of one to five milligrams are used, containing 25 to 100  $\mu\text{g}$  of the element of interest. Where possible, analytical protocols are devised to minimise sample preparation and fully exploit the automation.

SCRI also has expertise and resources for sample preparation from a wide range of matrices. These include plant sample drying and grinding, freeze drying and weighing facilities. Research support is aimed at developing new methods to assist the Institute's research programme.

**Organic Mass Spectrometry** The Institute's three state-of-the-art mass spectrometers, which are devoted to structural analysis of organic compounds, continue to yield valuable information on a diverse range of materials pertinent to the research remit of the institute. The core instrument is a Hewlett Packard 5989B MS ENGINE research-grade quadrupole instrument with electron impact, chemical (positive/negative) ionisation modes and a mass range of 2000 amu. Distributed processing software permits off-line data processing and reduces analysis times. This instrument can provide mass and structural data on a wide range of organic compounds.

A further bench top instrument is dedicated principally to the analysis of naturally occurring volatile com-

pounds. This consists of a Perkin Elmer automated thermal desorption system (ATD) linked to a VG TRIO-1000 quadrupole gas chromatograph-mass spectrometer and permits detailed characterisation of the profiles of organic volatiles generated by biological systems.

A Finnigan SSQ 710C dedicated liquid chromatography-MS instrument, with atmospheric pressure chemical ionisation (APCI) and electrospray ionisation (ESI) interfaces, is also available. This is suitable for samples whose high molecular weight, lack of volatility or polarity, make HPLC the preferred separation method. APCI and ESI are soft ionisation techniques and generally only produce molecular ions, e.g.  $[M-H]^+$  or  $MH^+$ , but the multicharge ionization mechanism of electrospray can extend the basic 2000 mass range of the instrument by a factor of about 20, giving a mass range of greater than 40,000 amu. This permits accurate mass determination of peptides, proteins and nucleic acids to within 0.1%, compared to the 5.0% error usually expected from SDS-PAGE determination.

Mass spectrometric analysis at SCRI covers a broad spectrum of chemical investigations generated by the research programme of the Institute. A wide range of plant metabolites has been analysed, both in the native form and as derivatives, including sterols, monoterpenes, sesquiterpenes, pentacyclic triterpenes, dimeric forms of phenolic acids, glucosinolates, long-chain wax esters, peptides, essential oils, carbohydrates, polychlorinated biphenyls and lipids, including fatty acids. The facilities are operated by experienced and expert staff, ready to tackle and solve most structural problems.

During 2001, SCRI took delivery of two new mass spectrometers to substantially increase its capabilities and sample turnover in both high throughput metabolic profiling and proteomics. The first of these is a ThermoQuest LCQ-DECA, an ultra sensitive ion-trap LC-MS<sup>n</sup> system. It is capable of many more scan functions than the existing SSQ710C spectrometer, including data-dependent full scan MS/MS, a tool of great utility in high throughput profiling. The same is true for protein/peptide sequencing, where rapid repeats of ion isolation and fragmentation generate



The new GC-MS and LC-MS system

sequence tags, which are searchable via on-board databases.

The other new instrument is a ThermoQuest TEM-PUS-TOF, an innovative GC-MS system capable of rapid detection, characterisation and quantification in fast GC separations. The benefits of the design provide parallel mass analysis with a short duty cycle at high transmission. This delivers rapid acquisition and fast sampling of narrow peaks at high sensitivity with high sample throughput.

A fundamental requirement of successful gas chromatographic mass spectrometric investigation is the development of robust chromatographic separations. Together with the MRS Lipid Analysis Unit, there are six gas chromatographs for high throughput analysis and the development of new separation protocols.