Analytical facilities

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Laboratory Accreditation

Within the Chemistry Department, 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 9002 by SGS Yarsely International Certification Services Ltd. 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 system are described in a Code of Practice document, a copy of which is issued to all members of staff. It 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. The Chemistry Department operates an electronic archival facility based on the use of a compact disc (CD) writer installed in a personal computer. 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, and two copies are made, one for the owner of the data and one for the archive.

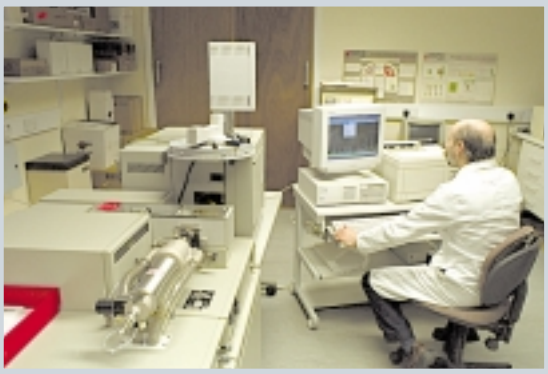
Stable Isotope Facility

Stable isotopes are now basic tools 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. With these, we can tackle most of the biologically important low atomic number elements, ^{13}C, ^{15}N, ^{18}O and ^{34}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 100µg 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 commissioned programme.

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 laboratory suite housing the instrument facilities will, in early 1999,



become part of a new integrated chemical facility which will include stable isotope mass spectrometry facilities, the MRS Lipid Analysis Unit and all of the Chemistry Department. 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 compounds. 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 characterization of the profiles of organic volatiles generated by biological systems. During the year, the gas chromatographic side was improved with the addition of a cold on-column injector. This should improve sensitivity of conventionally solvated samples and avoid undesirable degradation of thermally labile samples such as monoterpene alcohols.

A Finnigan SSQ 710C dedicated liquid chromatography-MS instrument, with atmospheric pressure chemical ionization (APCI) and electrospray ionization (ESI) interfaces, completes the facility. This has an ability to analyse samples whose high molecular weight, lack of volatility or polarity, precludes analysis on the other instruments. APCI and ESI are soft ionization 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.

The Institute's ISO 9002 certification now includes the analytical operation of both gas chromatography-mass spectrometry systems, with data archival facilities to appropriate levels, together with the required documentation, quality plan and standard operating procedures. Following a detailed survey and appropriate upgrading, we expect all of the important instrumentation to operate successfully at the start of the new Millennium.