Analytical facilities

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Laboratory Accreditation

Several laboratories within the Chemistry Department operate a formal Quality System, certified to BS EN ISO 9002 by SGS Yarsely International Certification Services Ltd. The stable Isotopes Facility and Lipid Analysis Unit of MRS Ltd were registered in December 1996, and the Gas chromatograph-Mass spectrometry Laboratories were registered in June 1997.

A Quality Plan for a generic Quality System for SCRI has been adopted and will be implemented throughout the Institute. A copy is included in the Institute's Corporate Plan. The Quality Plan 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 electronic archival facility has been set up within the Chemistry Department, 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 at maximum transfer rates, a CD can be written in less than 40 minutes. Two copies are made, one for the owner of the data, 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 100mg 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 sample types. 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 is particularly well equipped in the field of mass spectrometry (MS), with three state-of-the-art instruments devoted to structural analysis of organic compounds. Housed in a purpose-built laboratory suite, all systems have integrated computer control, library search capabilities and distributed data processing facilities. The core instrument is a Hewlett Packard 5989B MS ENGINE research-grade quadrupole instrument. The mass spectrometer has electron impact and chemical (positive/negative) ionisation modes with a mass range of 2000 amu, together with an autosampler and distributed processing software which will permit off-line data processing and reduce operating costs. It also has a particle beam liquid chromatography (LC)/MS interface which will complement existing LC/MS instrumental techniques. This instrument can provide mass and structural data on a wide range of organic compounds.

In addition, a bench top instrument is dedicated to the analysis of organic volatiles. This consists of a Perkin Elmer automated thermal desorption system (ATD) linked to a VG TRIO-1000 quadrupole gas chromatograph (GC)-MS and permits detailed char-

acterization of the profiles of organic volatiles generated by biological systems.

A Finnigan SSQ 710C dedicated LC-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 fatty acids. The facilities are operated by experienced and expert staff, ready to tackle and solve most structural problems. They are actively seeking full laboratory accreditation status and working practices are commensurate with recognised standards.

During 1997, the Institute's ISO 9002 certification was extended to include the analytical operation of the two GC/MS systems. This involved the successful in-house updating of instrument computer and archival facilities to appropriate levels, together with the required documentation, including a quality plan and standard operating procedures.