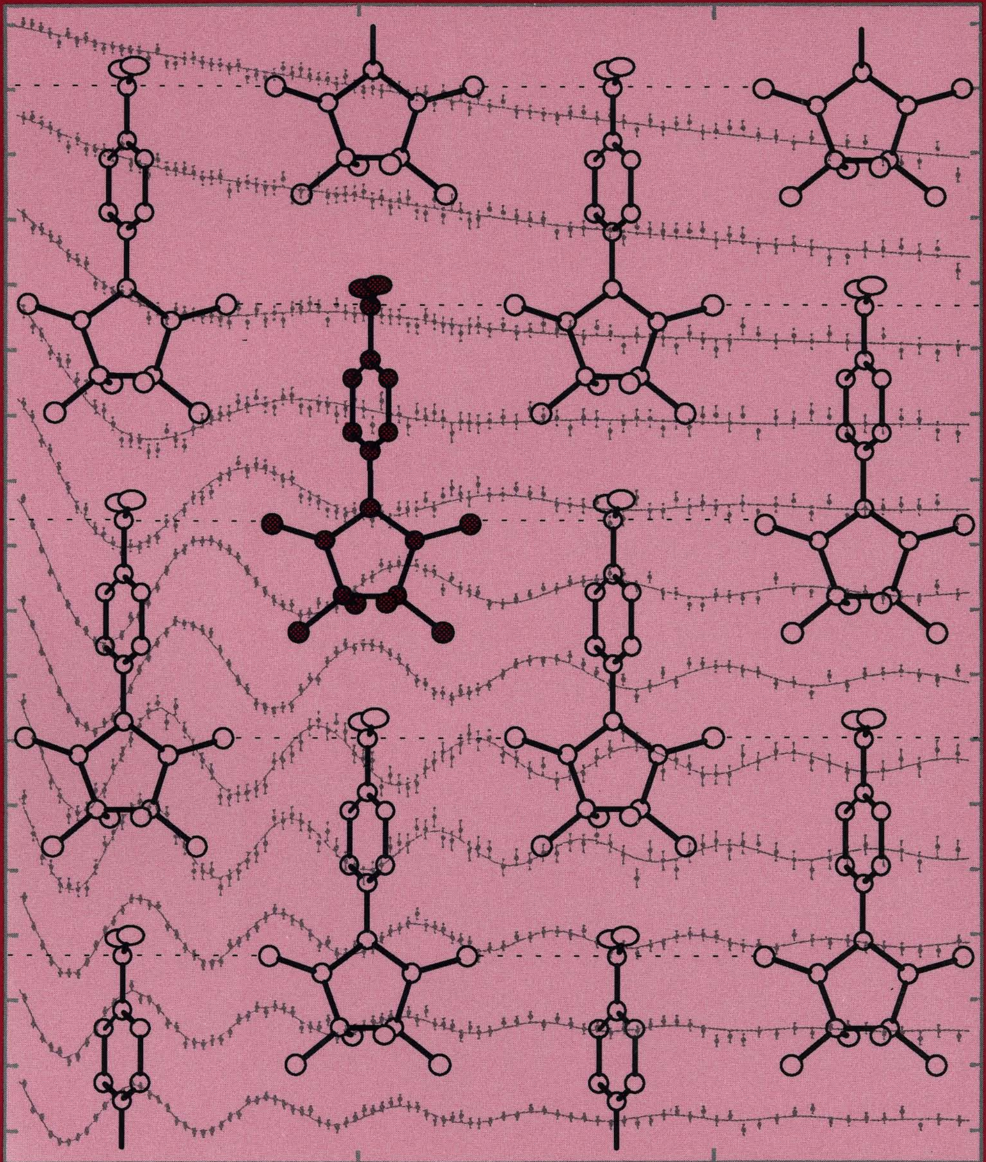
ISIS

1994



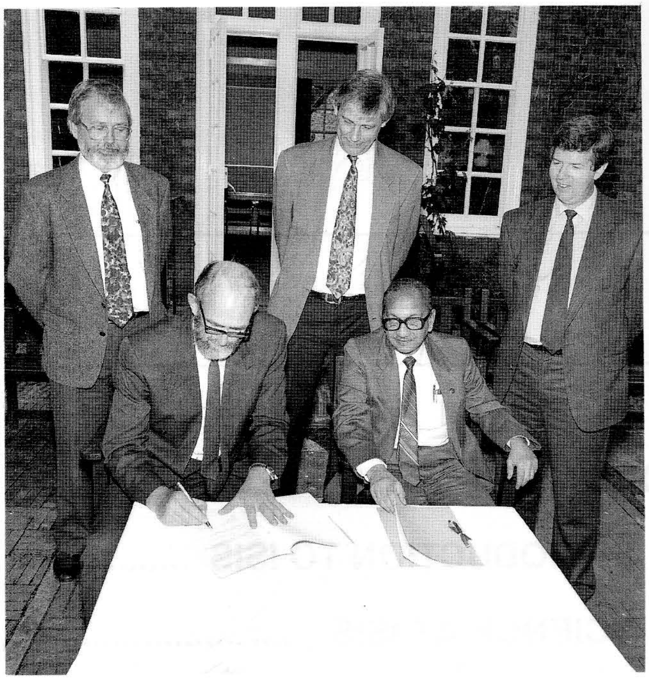
FOREWORD

The past year has seen many changes. The UK Government published a White Paper Realising Our Potential which set the scene for the science base to underpin wealth creation and contribute to the quality of life. As a result, the Research Councils have been restructured and have emerged with new names and new missions whilst we at RAL have merged with our sister laboratory at Daresbury to form DRAL, the Daresbury and Rutherford Appleton Laboratory. Through all these changes, both the basic and strategic science programmes at ISIS have continued to expand, and the prospects for the future have continued to improve.

Once more, we have had our most successful year to date. ISIS produced 10,000 μA-Instrument-hours of beam, and, as can be seen from this Annual Report, excellent science continues to flow from the instruments on the neutron and muon sources as well as from the neutrino experiment KARMEN.

The present phase of development projects are now bearing fruit: the CEC-funded muon beamlines are now fully operational; the development of engineering science on the TEST beam is well advanced and the Japanese-funded RIKEN complex for muon catalysed fusion research is on schedule to receive its first beam in the autumn, as will SURF, the first of the Collaborative Research Grant funded neutron instruments. Future expansion of the programme at ISIS is now guaranteed through this CRG mechanism with four major projects all receiving substantial development funds: MAPS (Warwick/Oxford/AT&T); OSIRIS (Salford/India); LOQ (Reading) and high pressure cells (Edinburgh/Paris). We also welcome for the **first time** at ISIS a significant nuclear physics presence as the RIST project begins to gather momentum.

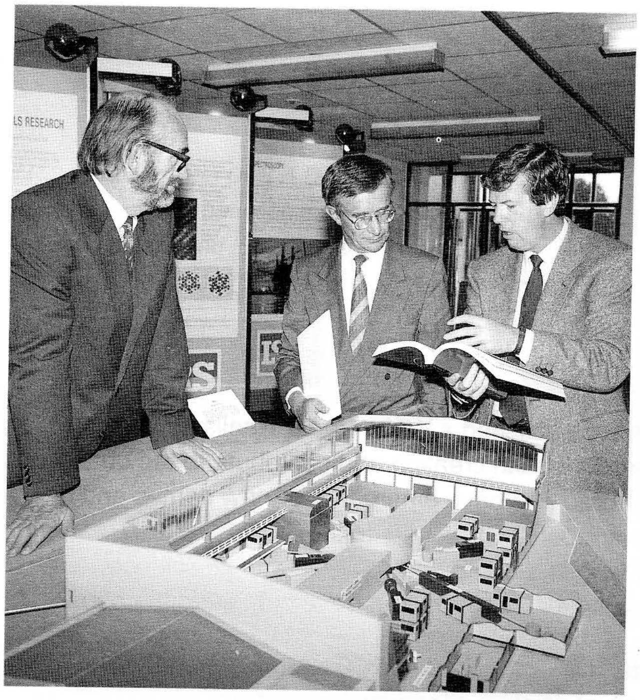
The results of the recent National Audit Office report on ISIS could hardly have been more impressive; our sincerest thanks to those who took part in the survey - and told the truth! For the first time we have had a totally independent measure of the level of user satisfaction at ISIS, and even though it was extremely high, we will nevertheless strive to improve the service we offer to our user community.



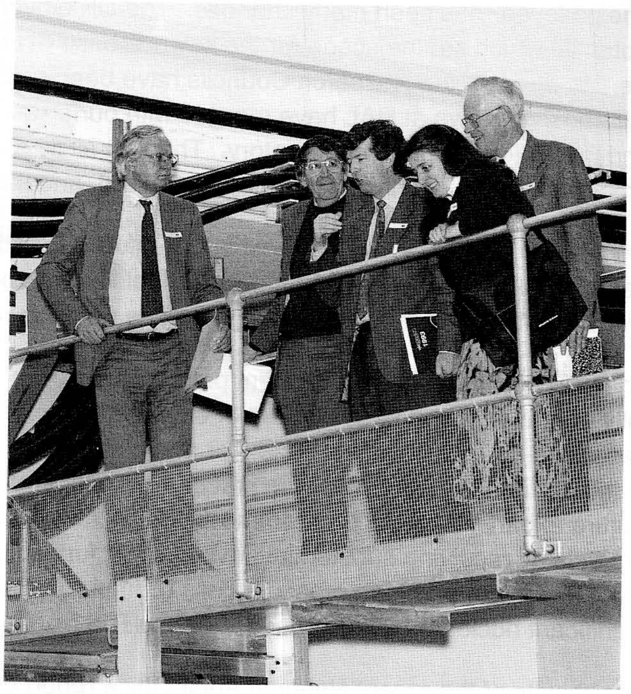
Dr Paul Williams and Dr B A Dassannacharya sign the second agreement between the Indian Bhabha Atomic Research Centre and ISIS, watched by Dr Colin Carlile, Professor Keith Ross (Salford) and Dr Andrew Taylor. *[94RC3529]*

Professor W Gläser talks to Dr Andrew Taylor during the HCM assessors visit. *[94RC2616]*

Dr A E Hughes and Dr J Borgman signing the EPSRC-NWO agreement extending the Dutch support for ISIS. *[94RC1969]*



(left) Austrian Ambassador Hamburger is introduced to ISIS by Paul Williams and Andrew Taylor during a visit by representatives of the Austron project. *[93RC4104]*



(right) Professor Chris Dobson , Professor Laurie Challis, Andrew Taylor, Irene Scullion and Professor Dennis McWhan during the Facilities Commission visit. *[94RC1133]*

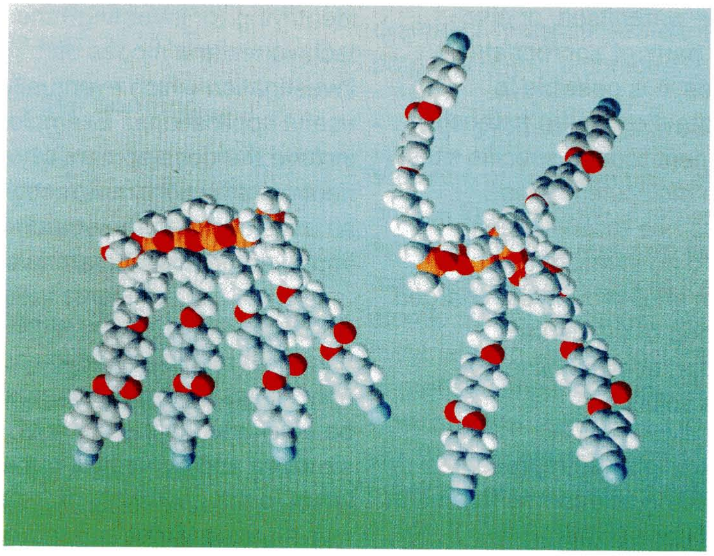
(left) Uschi Steigenberger extolling the virtues of PRISMA to Professor Eugenio Tabet, the Italian Scientific attaché. *[94RC3035]*

(right) Richard Ibberson and Mike Johnson discuss the structure of $(Ca,Y)F_{2+x}$ *with Mr Tom Casey (Ireland) and Dr Peder Larsen (Denmark) during the HCM assessors visit.* *[94RC2619]*

transitions to be studied, often in the quest for 'pure' knowledge, but the **same** parameters allow the study of complex zeolite or drug materials.

In general, much of the industrially-focused work at ISIS can be split into three categories:

- **Applied research** that is aimed at solving a particular commercial problem (e.g. in formulating new refrigerant liquids to replace CFCs);
- **Strategic studies of materials** whose properties will be crucial to developing new products and techniques in the future, e.g. electrocatalysts, superconductors, ceramics);
- **Development of new techniques** which will assist industrial problems in the future, for example the combination of neutron diffraction and computer simulation in liquids scattering and the development of detailed isotopic labelling schemes enabling surface structure to be determined with unprecedented resolution.



The configurational changes in adsorbed polymers can be followed using neutron reflection.

ISIS and the future

The breadth and depth of the science programme clearly shows that ISIS is a resounding success. Having reached its design parameters, producing 200 µA of 800 MeV protons, ISIS is **the** most powerful spallation neutron source.

Just as significant as the past successes, however, are the opportunities in store. ISIS is still a young source - spallation neutron sources are relatively underdeveloped, with much potential to realise. As an example, ISIS was conceived without a surface science programme. The insights provided by the surface reflectometer CRISP into areas as diverse as detergents and magnetic films have established time-of-flight reflectometry as a significant new technique impacting upon industrial applications as well as basic surface science. Similarly, in 1984, high temperature superconductivity and fullerene research did not exist. These two waves of condensed matter research, unplanned and unforeseen, have dominated the scientific literature over the past eight years. Research at ISIS has played a key world role in both these areas, providing definitive results on both the structure and dynamics of these important materials. With this evidence, it is clear that as new materials are discovered and engineered ISIS will continue to play a leading world role in key areas of research. Moreover, the technique developments at ISIS over the past decade open up further opportunities. Surface reflectometry has been cited as one new development. Experience at ISIS, however, has led to a completely new appraisal of the potential of cold neutron research at spallation sources. Conventional wisdom held that the intense epithermal neutron spectrum would be the principal advantage of spallation sources over reactors (this is indeed the case) while cold neutron research would, relatively, be compromised. It is now clear that intense cold neutron fluxes **can** be produced at ISIS and opportunities for substantial further enhancement exists in the construction of a second low frequency target station. This will be a crucial development area for spallation neutron sources, permitting new large scale chemical and biological science to be performed. It is also a necessary development step for the next generation of spallation neutron sources, such as the proposed European Spallation Source, ESS, that will lead neutron research in the next century.

The future of neutron scattering is indeed bright, and at ISIS we look forward to playing a full part in it.

Tunnelling Spectroscopy

The rotational tunnelling of methane molecules on the (100) surface of MgO were investigated. This system is of particular interest due to the commensurate square lattice surface structure. Previous measurements have shown the methane to be adsorbed in a dipod configuration with two hydrogen atoms binding to the substrate. When one of the H atoms is replaced by a deuteron, however, the spectrum changes from a multiple peak to a single peak spectrum at ~135 μeV (Larese, **A302**). The geometrical attitude of the molecule is now being re-examined.

There were also measurements of the tunnelling spectra of the ammonium rubidium halides under varying conditions of both temperature and pressure (Mukhophadhyay, **A305**). There is found to be remarkable similarity to the effects observed in the methane/rare gas systems, with trends in spectral degradation with methane/ ammonium ion concentration and rotor energy with host lattice size mimicking each other closely.

The effect of partial deuteration was investigated in a series of measurements of methyl group-containing molecules, with a view to understanding rotor-rotor coupling (Fillaux, **A299**). The results obtained to date undermine the coupled pair model and yield further support for the collective rotation model (sine-Gordon dynamical theory).

The isolated molecule of tribromomesitylene has three-fold symmetry and three equivalent methyl groups, but the inelastic neutron scattering spectrum exhibits three distinct tunnelling lines as a consequence of the low (triclinic) crystal symmetry. The spectrum from this material was measured from a single crystal as a function of angle, allowing the unambiguous assignment of the transitions to individual methyl groups (see **Highlight**).

Methyl group Tunnelling in a Single Crystal of Tribromomesitylene

Tunnelling spectroscopy is a sensitive probe of the local potential experienced by small molecular rotors such as $-CH_3$, CH_4, NH_3 and NH_4^+, particularly when coupled with the measurement of librational modes. In particular, details of intra- and inter-however, a fundamental problem is encountered since it is not possible to relate specific tunnelling lines to specific librational modes originating from crystallographically inequivalent groups. Attempts have been made, for example in the case of toluene, to make such an assignment by studying the temperature dependence of the line width which, according to theory, shows an activation energy related to the librational energy. This is fraught with difficulty. Another approach is to choose a material in which the occurrence probabilities of the inequivalent groups is different, and this has been done in the case of tetramethyl lead, where the occurrence probability, and hence the line intensities, of the two different methyl groups is 3:1.

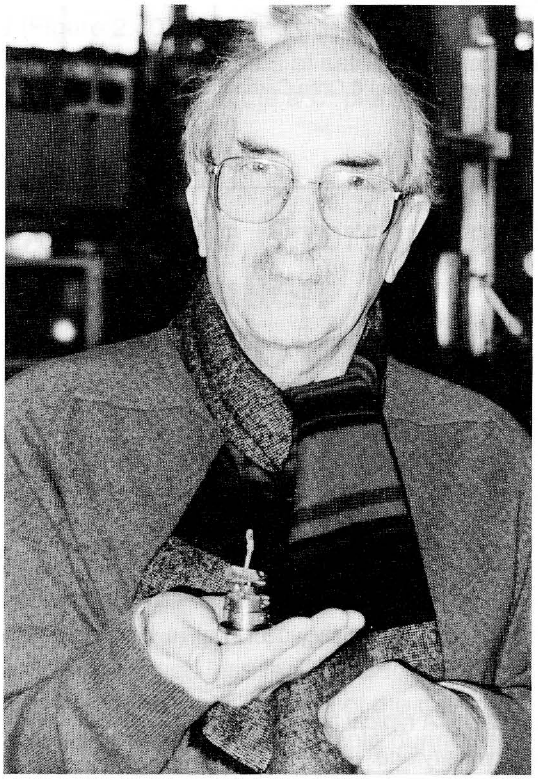


Figure 2.37 *Professor Jean Meinnel of The University of Rennes is shown with the 165 mg single crystal sample of tribromomesitylene.*

INVESTIGATIONS OF SUPERCONDUCTIVITY IN RARE EARTH-Ni_2B_2C

Contributed by ***Brian Rainford*** *(Southampton) and* ***Bob Cywinski*** *(Reading)*

The discovery of a new family of superconductors always arouses considerable interest. Such interest has been particularly intense in the case of the new RE-T-B-C quaternary compounds, where RE is a rare earth or Y and T is Ni or Pd. This is largely because superconductivity is observed at temperatures close to the maximum yet found for metallic alloys, with a T_c of 23 K being reported for Y-Pd-B-C. However, early claims that these compounds constitute a new class of superconductors still require substantiation. Investigations of these claims, and characterisation of the superconducting ground state of RE-Ni_2B_2C have been carried out on the POLARIS, HET and MuSR instruments.

Neutron powder diffraction

The crystal structures of RE-Ni_2B_2C with RE = Er, Ho and Y, were determined at 300 K using the POLARIS diffractometer. The samples were found to have a modified $ThCr_2Si_2$ structure, space group I4/mmm, with the carbon atoms occupying interstitial sites between the boron planes. Rietveld refinements of temperature dependent (2 - 40 K) diffraction patterns showed a very small thermal expansion and no evidence of a structural transition close to T_c. The crystal structure of YNi_2B_2C is shown in Figure F10.1.

Evidence for the coexistence of magnetic order and superconductivity was found in the diffraction pattern of $ErNi_2B_2C$ (T_c = 10.5 K). The appearance of additional peaks at low temperatures can be attributed to an incommensurate antiferromagnetic structure, with spins constrained to the *ac* plane and characterised by a (Q,0,0) modulation where Q = 0.5594 ($2\pi/a$). The temperature dependence of the (Q,0,0) reflection at d = 6.31 Å is shown in Figure F10.2, indicating a Néel temperature of 6.5 K.

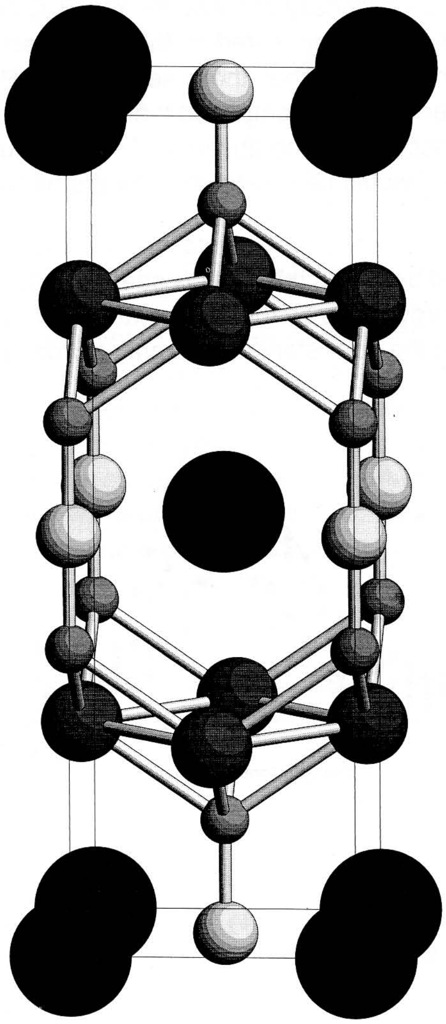


Figure F10.1 *Structure of* YNi_2B_2C.

MUON BEAM DEVELOPMENTS

EC Surface Muon Facility

1994 saw the completion and successful commissioning of the upgrade of the ISIS surface muon facility funded by the CEC Large Installation Plan. This upgrade consists of the following principal components:

- Use of a fast E-field septum kicker to separate and divide the two muon pulses into three beam lines, thereby providing single muon pulses at 50 Hz to apparatus situated in each experimental area;
- Provision of remote collimation in each beam-line to allow tailoring of the muon spot size to the individual experimental requirements;
- Provision of a new longitudinal high field (0.4 T) μSR spectrometer in the second area designated EMU, in addition to the original μSR spectrometer retained in the MuSR area;
- Provision of a third experimental area, designated DEVA, for the development of pulsed μSR techniques and muonium studies with pulsed lasers.

The facility (Figure 3.1) was completed by early June 1993 and essential commissioning carried out in a period of three days at the start of the cycle. The calculated parameters of the new beam lines and the E-field kicker were verified in practice and the distribution of single muon pulses to the three areas was demonstrated.

The new facility at ISIS using fast kicking of a pulsed muon beam is the first in the world to successfully demonstrate these features. It utilises optimally the double pulse structure of the ISIS extracted proton beam and allows simultaneous operation of all three experiments with 50 Hz muon pulses.

In addition to the successful commissioning of the E-field kicker which is used in the normal operational mode of the facility, delivery of all the available muons to a single area was demonstrated using the two switchyard magnets installed in the new beam line.

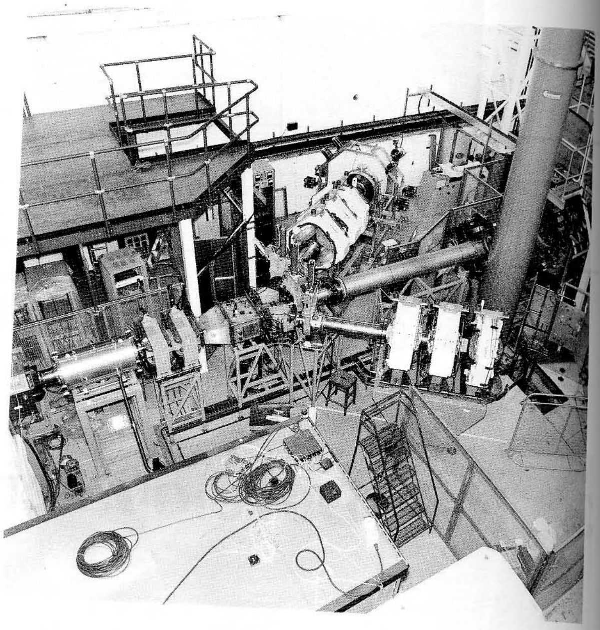


Figure 3.1 *The EC Surface Muon Facility.* *[93RC2479]*

The RIKEN-DRAL Decay Muon Channel

The RIKEN-DRAL decay muon channel (Figure 3.2) is entering the final stages of construction with an expected commissioning date of October 1994.

The superconducting solenoid constructed by Mitsubishi was successfully commissioned during August 1993 and found to perform as expected.

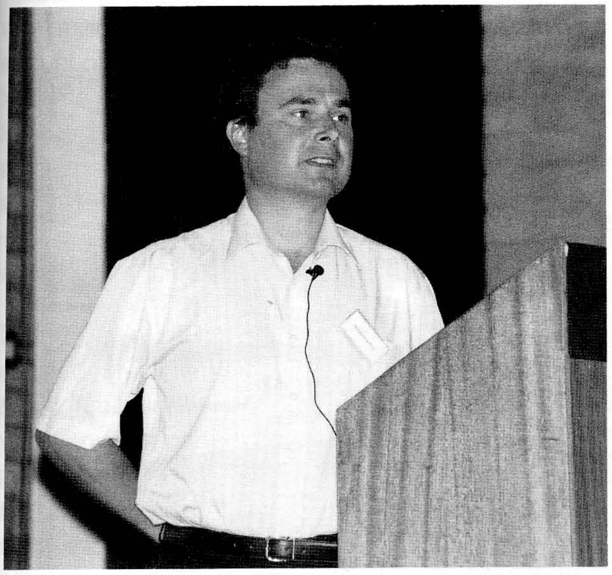
The muon extraction system is now being assembled with a total of eighteen quadrupole magnets (Figure 3.3). A number of key components from Japan have been or will shortly be delivered, including the fast magnetic kicker. This has been tested in Japan and meets the stringent operational requirements for switching the two muon pulses into two separate beam lines up to a muon momentum of 55 MeV/c. Other Japanese equipment installed include the septum magnet, two electrostatic separators to remove positron and electron contamination, and a pair of small switchyard magnets. These latter components will be used to direct both muon pulses to a selected experimental area for muon momenta above 55 MeV/c up to a maximum of 120 MeV/c.

'bottleneck'. The invited participants came from the UK, Denmark, Japan and America, all experts in single crystal inelastic measurements. Discussions included the need for a standard, European wide neutron data format, the tools to visualise large data sets, and the likely transformations required for MAPS raw data. Work is now starting on these problems, with the ensuing software to be tested with HET and MARI results.

SURF - Surface Reflectometer

The new reflectometer, SURF, is funded through a grant awarded to a CRG group headed by Rob Richardson (Bristol) and Adrian Rennie (Cambridge), along with a component from the Australian contribution to ISIS.

The reflectometer design has been optimised for applications in surface chemistry and SURF will have a major impact on the advancement of the investigation of adsorption of the liquid-solid and liquid-liquid interfaces and in the study of complex multi-component systems. The enhanced flux, beam focusing at the sample, and the area detector should substantially enhance the facilities available for the study of chemical interfaces.



Rob Richardson, one of the grantholders for the SURF project, speaking at the 1993 NBUM meeting at Sheffield.

Figure 3.8 *Peter Phillips installing the intermediate shielding wall for SURF. [94RC5044]*

SURF is now under construction (Figure 3.8) and commissioning should commence in October this year.

OSIRIS - Cold Neutron Spectrometer and Diffractometer

The realisation of the power of cold neutrons on pulsed sources, particularly for high resolution measurements, has led to the funding of the OSIRIS project to map out the potential of cold neutron instrumentation. The OSIRIS project (Figure 3.9) will be built in three phases.

The first phase will be funded by BARC Bombay and will involve extracting a second independent neutron guide from the IRIS beam hole (see **Chapter 6**).

The second phase will be funded by EPSRC via a Collaborative Research Grant to the University of Salford (Professor Keith Ross). This will enable the neutron guide to feed neutrons of wavelengths up to 30 Å to a 2.15 m diameter analyser tank at 33 metres from the moderator, in which a large diffraction detector will be placed. The neutron guide will be provided by PSI Switzerland and will be supermirror coated, providing count rates which

Figure 4.6 *The sample preparation laboratory in R55.* *[94RC4535]*

Part of the support role is to provide laboratory areas fully equipped with standard equipment for user sample preparation (Figure 4.6) and these facilities have been substantially improved this year. Together with these responsibilities goes safety, both general beamline safety such as the provision of interlocks and radiation monitors, and sample and sample preparation safety to COSHH regulation standards. Remarkably, the group made safety assessments on some 5000 samples last year.

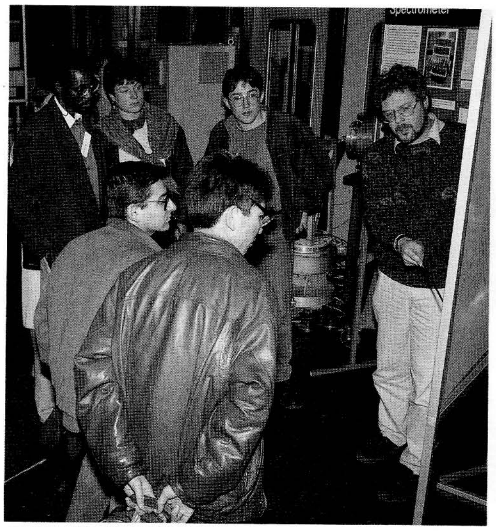
In addition the instrument operations group is also responsible for maintenance and modifications to existing beamlines and the installation of new beamlines, requiring active involvement at both the instrument planning stage and during installation.

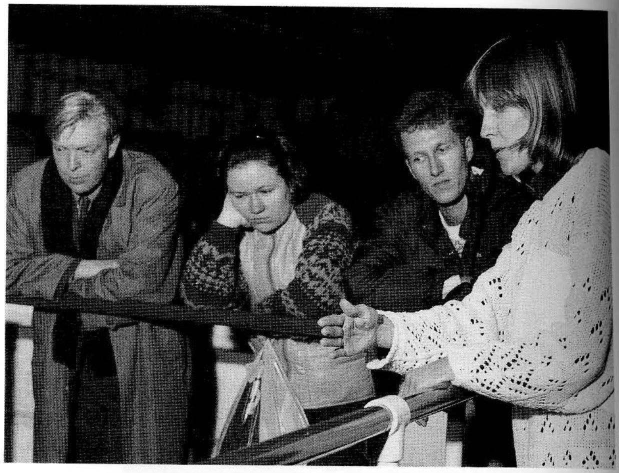
Last year activities in this area included:

- Installing a new sample tank on POLARIS:
- Installing shielding and an argon filled flight path in SANDALS;
- Specification and procurement of beam line defining apertures and their associated control electronics for HRPD, MARI, SANDALS and HET;
- An electrical noise survey of the experimental area;
- Planning of SURF installation.

A further initiative has been the specification of replacement hardware for the CAMAC sample environment and instrument control systems. The assessment of the requirements for such a system has been carried out in conjunction with representatives from the instrument scientists, computing and sample environment groups.

The operations group, enjoying the sunshine. *[94RC4136]*

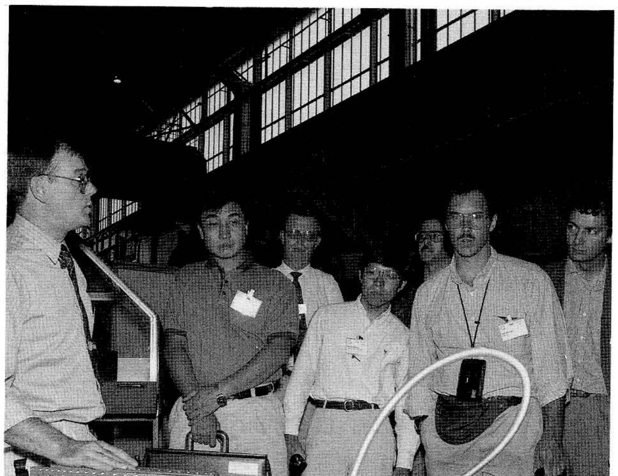


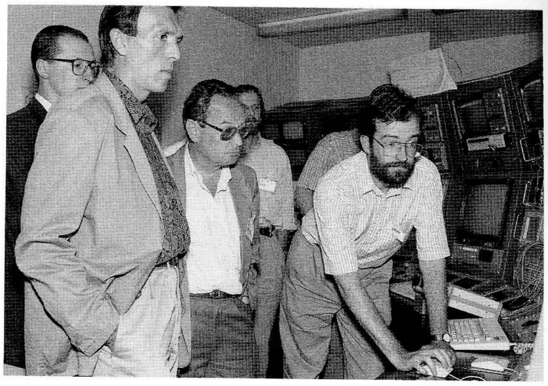


Students on the IOP Magnetic Measurement Techniques Course learn about ROTAX from Holger Teitze (top left)... *[93RC4864]*
...and the muon facility from Sue Kilcoyne (above). *[93RC4832]*

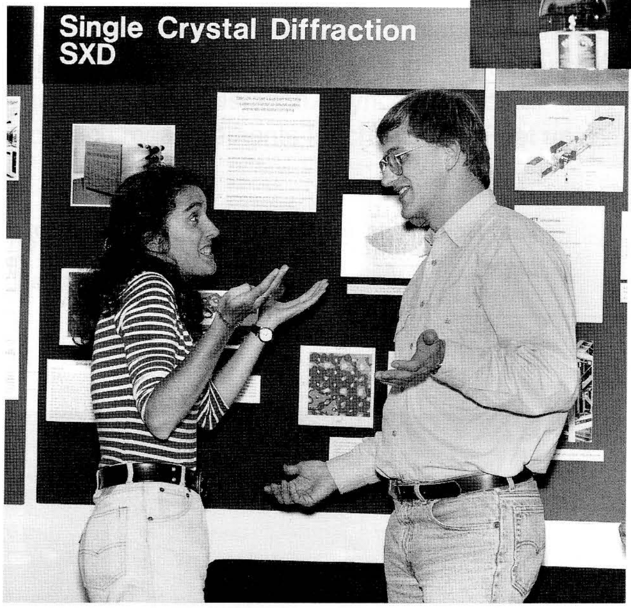
(left) Richard Heenan showing a group of sixth form students how to operate the ω rotation CCR. *[94RC3602]*

Delegates at the EPAC conference are introduced to ISIS by (clockwise from below) Gavin Williams at the ISIS model, Bob Mannix in the Main Control Room and Bob Hall in the Target Services Area. *[94RC4155], [94RC4154] & [94RC4162]*





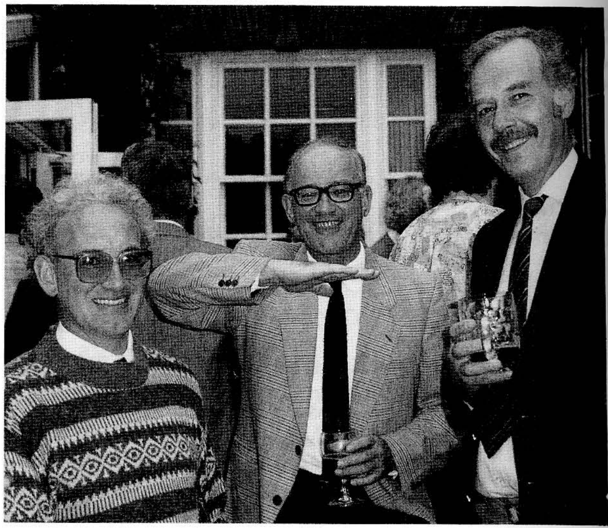
Randal Richards (Durham), Chair of NBC, doing it his way at the Sheffield NBUM meeting.



Mutual incomprehension, but Mireri Antxustegi (San Sebastian and Strathclyde) and Toby Perring seem to be enjoying the conversation in any case.

[93RC4144]

Andrew Taylor and Paul Williams in Korea, determined to reverse the normal trend by exporting ISIS useage to that expanding Far Eastern market.



ICANS
twelve

*Delegates at **ICANS XII**. Clockwise from top left:*

Tasso Springer and Günter Bauer

Rob Robinson, Herbert Lengeler and Tim Broome

Devinder Sivia, Phil Seeger Joyce Goldstone

Rob Robinson and Jack Carpenter

Myriam and Paul Williams, Bruce Brown and Roger Pynn