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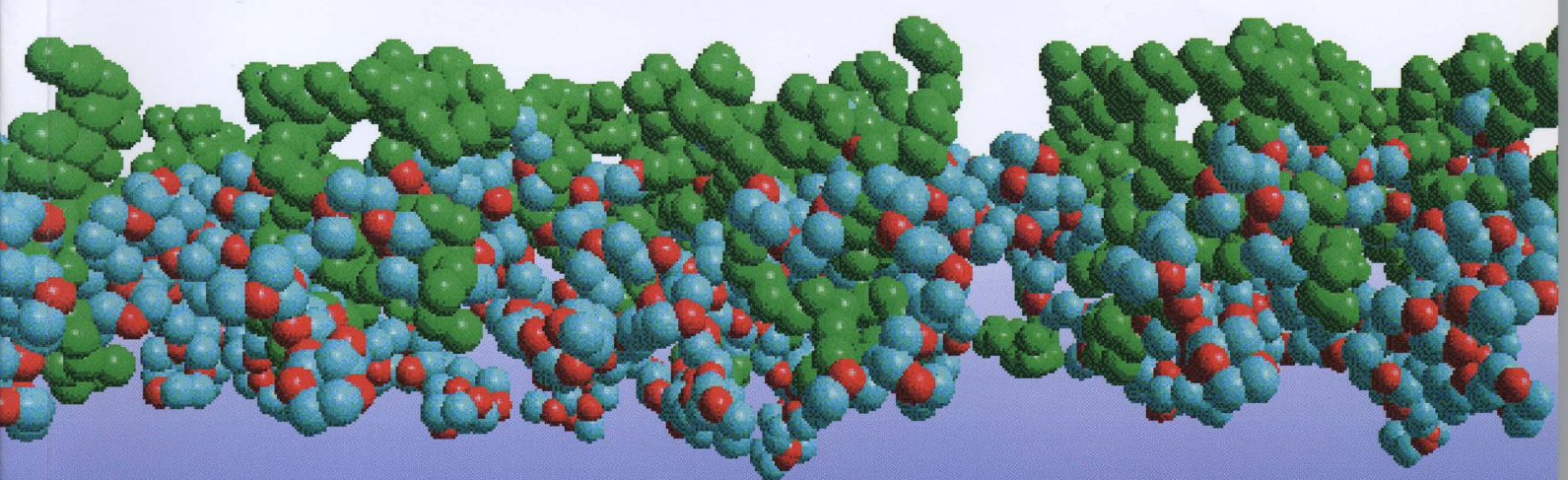
COUNCIL FOR THE CENTRAL LABORATORY
OF THE RESEARCH COUNCILS

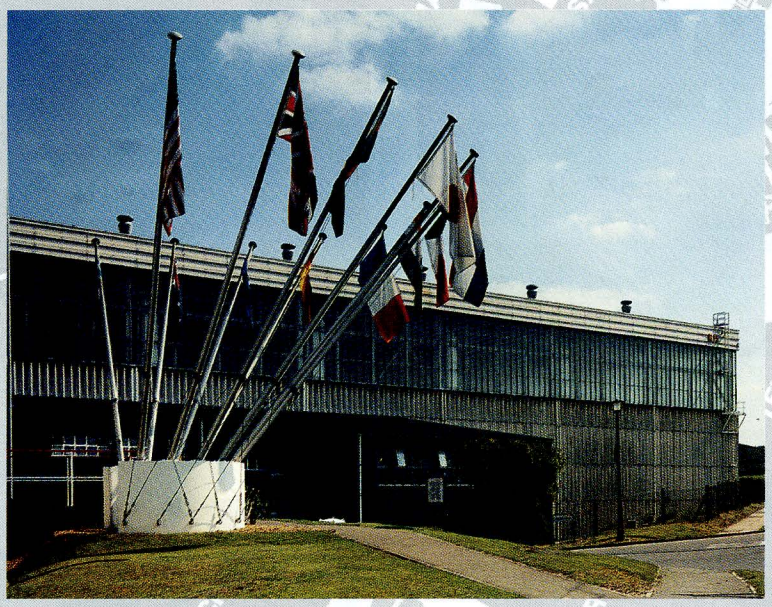
ISIS 97

The Rutherford Appleton Laboratory, ISIS Facility Annual Report 1996-97



ISIS

Foreword



After an inauspicious beginning to the operating year, ISIS bounced back with improved reliability once again to achieve - and exceed - its 200 μA design current and in the end to deliver only marginally less integrated current than last year's record. The quality and breadth of the scientific programme at ISIS continues to be outstanding and the number of publications continues to grow. The volume of ISIS Experimental Reports has grown so large that the only practical way to issue them is as a CD - inside the back cover of this Report !

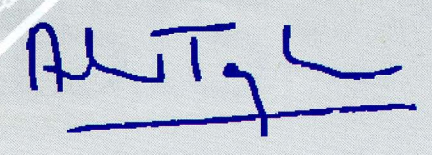
Working with the user community both at home and abroad, we have succeeded in providing significant technical enhancements to our instrument suite. The Large Scale Structure community are benefiting from the complementary nature of CRISP and SURF; POLARIS has been successfully moved and upgraded; the pressure programme continues to expand; MARI will shortly have its complete complement of detectors and the successful commissioning of a position sensitive array on HET bodes well for MAPS; the twin IRIS-OSIRIS guides are installed and engineering design and manufacture proceeds apace on the next generation of ISIS instruments OSIRIS, MAPS and GEM.

The ISIS community has made good use of new funding available from the Higher Education Funding Council - the Joint Research Equipment Initiative - where bids from Kent, Warwick, University College London and Oxford secured a new instrument for chemical spectroscopy - TOSCA (with support from Italy), an enhancement to MAPS (with support from the USA), the development of a cold muon facility and the much needed biology support facilities (again with help from Italy).

The highly successful UK-Japan Collaboration which gave us MARI and catalysed advances in coherent excitations studies at pulsed neutron sources was formally renewed for ten more years in January at a simple ceremony in Tsukuba. One of the immediate benefits of this next phase will be strong Japanese involvement in MAPS and a close co-operation with the scientists and engineers working towards the realisation of the Japan Hadron Facility.

UK research groups using ISIS have responded well to the radical access mechanism introduced by EPSRC earlier this year and there is a healthy demand for all instruments at ISIS. International partners and UK Research Council grant holders are assured of access to the ISIS scheduling system, as are outstanding proposals independent of their origin. In addition, limited transitional arrangements will remain for UK groups actively seeking research council support in the coming year.

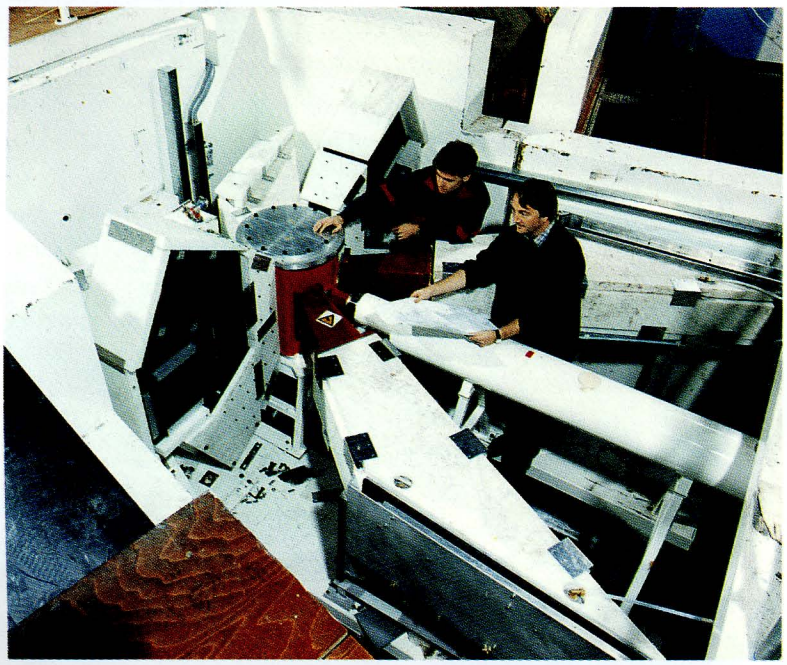
The future of the facility depends on the success of this mechanism: it is up to all of us - in the facility, in the community and in the Research Councils - to do our best to make it work.



Crystallography

The crystallography programme at ISIS is principally carried out on the two powder diffractometers HRPD and POLARIS and the single crystal instrument SXD. In addition, in the past year a user programme in high pressure diffraction has been established on the PEARL beamline. These instruments offer a combination of high flux and high resolution and cover the full range of crystallographic science. The ultra-high resolution of HRPD allows phase transitions, subtle structural effects and complex structures to be studied; the high flux medium resolution POLARIS offers rapid data collection from powder samples, specialising in the study of materials in complex sample environments and offering opportunities in *in situ* and time-dependent studies; the single crystal diffractometer SXD exploits the time-of-flight Laue technique in structural and diffuse scattering applications and the high pressure component of the PEARL programme utilises the Paris-Edinburgh cell to offer facilities for powder diffraction under very high pressures, combined with a low temperature capability. Crystallography is also undertaken on a range of other ISIS instruments including LAD, ROTAX and IRIS.

In the past year POLARIS has been moved to its current location on beamline N7. The move was achieved with a minimum of disruption to the user programme and the performance of the new instrument meets or exceeds expectations in every area. With improved count rate and resolution on offer, depending on the sample being studied, POLARIS is now well placed to remain in the forefront of high flux neutron powder diffraction in the future. Looking further ahead, the CRG-funded GEM diffractometer will offer still further opportunities to exploit new and exciting applications of materials diffraction.



Ron Smith and Steve Hull during the relocation of POLARIS to N7 [96RC5193].

HRPD

The uniquely high resolution available on HRPD for powder diffraction studies continues to offer exciting possibilities across a wide-ranging user programme.

High resolution is often associated with long counting times. However, a number of recent experiments have demonstrated the success of HRPD in carrying out detailed and comprehensive "structural surveys" with sub 10-minute counting times and small temperature increments. This is exemplified by phase transition studies on the negative-thermal expansion material ZrW_2O_8 (***Scientific Highlights, page 38***) where the underlying mechanism explaining the novel behaviour was elucidated following a 2 day experiment comprising 275 runs over a temperature range of 2K to 550K. Other notable studies of this type include the order-disorder transition in β-sulphur (**David, RB8555**) and in the canonical ferroelastic relaxor PMN ($PbMg_{1/3}Nb_{2/3}O_3$) (**Darlington, RB7765**).

In addition to traditional phase transition studies, material processing can be effectively studied *in situ* using the resolution and count-rate of HRPD. The annealing process of cubic $BaTiO_3$, at above 1000°C, to yield the technologically useful equilibrium tetragonal form at room temperature has been investigated (**Rosseinsky, RB7671**).

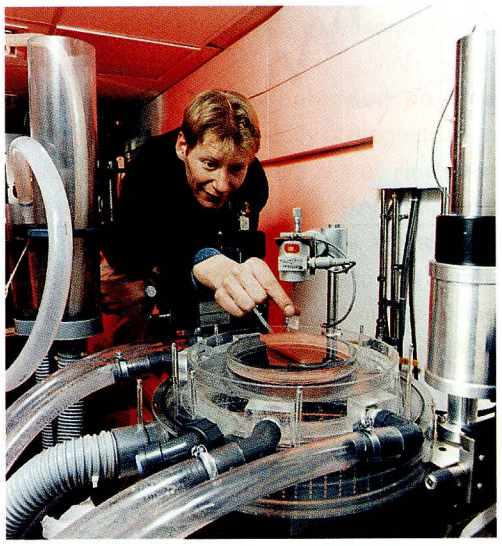
Large Scale Structures

Small angle neutron scattering, SANS, and neutron reflectometry are important techniques for the study of soft matter, complex fluids, surfaces, interfaces and thin films. On the small angle diffractometer, LOQ, and on the reflectometers, CRISP and SURF, there is an extensive programme in these fields. These instruments exploit the features of pulsed neutron sources: a wide dynamic range and the capability of good resolution; the selectivity of neutrons (through H/D contrast variation); the ability to access "buried" interfaces, and the use of complex environments.

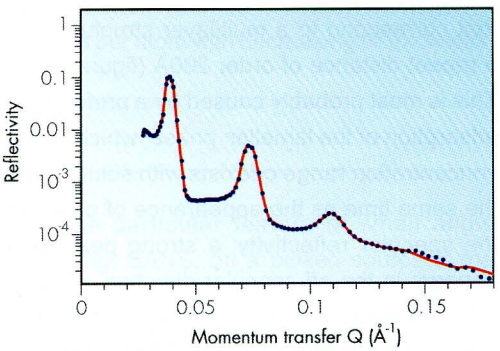
CRISP and SURF

The applications of reflectometry on CRISP and SURF cover a broad range of science, with a clear trend towards the study of complex multi-component systems and environments.

An overflowing cylinder has been developed by a group at Wageningen Agricultural University which allows the study of protein adsorption at the air-water interface in non-equilibrium conditions and access to timescales from milliseconds to minutes (**Boerboom, RB8548**). The results obtained have important implications for foam formation and stability. A high temperature furnace has also been developed to study metal-ceramic interfaces, allowing the study of industrially important wetting processes. *In-situ* electrochemistry is a further example of this trend (***Scientific Highlights, page 50***).



Frank Boerboom (Wageningen University) adjusting the overflowing cylinder, used on SURF to study the adsorption of proteins in non-equilibrium coconditions [97RC1219].



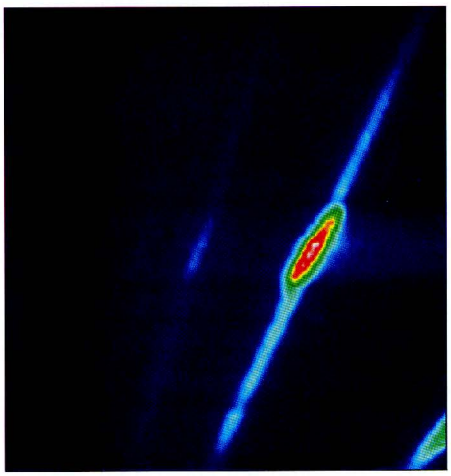


Figure 2.8. (a) Specular reflectivity for 2% AOT solvent in D_2O at 25°C. The solid line is a calculated curve for a damped bilayer structure extending from the interface into the bulk solution. (b) multidetector data for a 2% h-AOT in D_2O solution, at an angle of 1.5°.

Off-specular scattering provides information about inhomogeneities in the plane of the surface or interface, on a length scale which overlaps with light scattering, but with the added attraction of *in-situ* measurements at buried interfaces. Among experiments examining the off-specular scattering have been studies of optical gratings, as model systems for testing the theory and limitations of the neutron reflection technique (**Zarbakhsh, RB8832**), and studies of the surface of aerosol-OT surfactant solutions

Molecular Spectroscopy

The Molecular Sciences instruments group around a central interest in the forces which maintain molecular shape and ultimately dictate reactivity and functionality. These forces can be studied at several levels, from the fundamental interactions of quantised liquids through to the flexible arrangement of biological molecules.

TFXA

This year on TFXA has seen a growing interest in the dynamics of weakly constrained molecules and their fragments. This work is of considerable interest to the biology community and those working in related fields, like pharmacology. Although the biological activity of large molecules is largely controlled by their tertiary structure (shape), local flexibility endows these molecules with a conformal adaptability which allows them to take on different activities. Modern approaches to the understanding and exploitation of biological and pharmacological systems involve computational modelling of their conformation and dynamics. The functional form of the atomic interaction potential plays a fundamental role in these calculations. Current interest in biological calculations centres around the Lennard-Jones 12-6 potential but this has been found wanting in its ability to predict the weak interactions controlling tunnelling transitions in molecular systems. One such system is aspirin (**Johnston, RB8568**). The strong feature clearly visible in the INS spectrum of the compound (figure 2.16) is the fundamental transition of the methyl torsion motion, i.e. the same potential well as for tunnelling but over a different region. The softening of this transition with temperature occurs because of lattice expansion and should allow the strength of the interaction to be mapped as a function of distance between molecules. Similarly the change in dynamics of proteins upon folding has also been studied in Staphylococcal nuclease (**Kataoka, RB7588**).

The study of catalysts remains an important part of the TFXA programme. Copper based catalysts, which are used in the industrial synthesis of methanol, have been studied (***Scientific Highlights, page 64***)

Metal hydride complexes are of considerable interest for hydrogen storage applications. Simple metal hydrides are comparatively rare and also serve as model systems for hydrogen-in-metal materials and for homogeneous and heterogeneous catalytic processes. Studies of Mg_2FeH_6, Mg_2FeD_5H and Mg_2FeD_6 (**Yvon, RB7600**) have led to the development of an empirical forcefield, and suggest that there is a dynamic distortion of the perhydrido complex, an effect that is not observed in the deuterated species.

The arrival of TOSCA early next year, with a fivefold increase in detector area, will greatly enhance the potential for molecular spectroscopy studies.

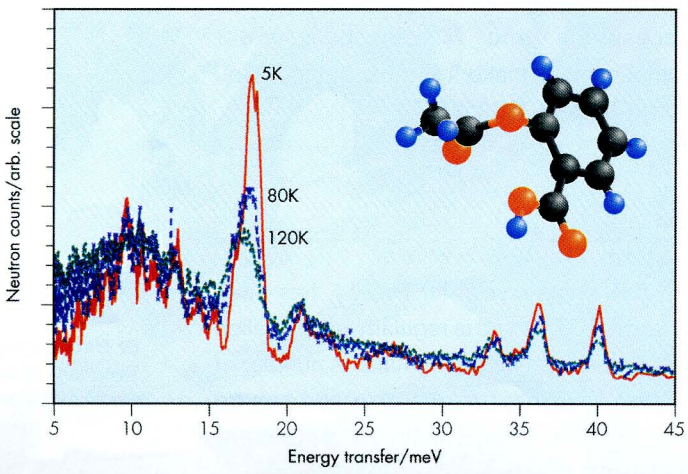
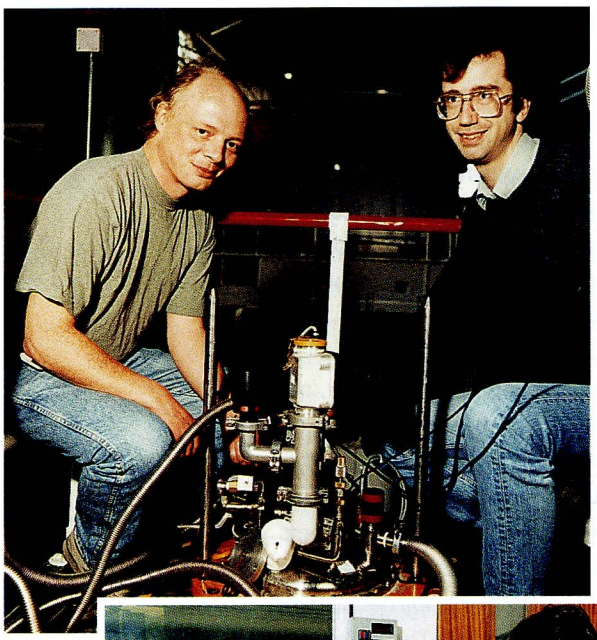


Figure 2.16. Inelastic scattering spectrum of aspirin.

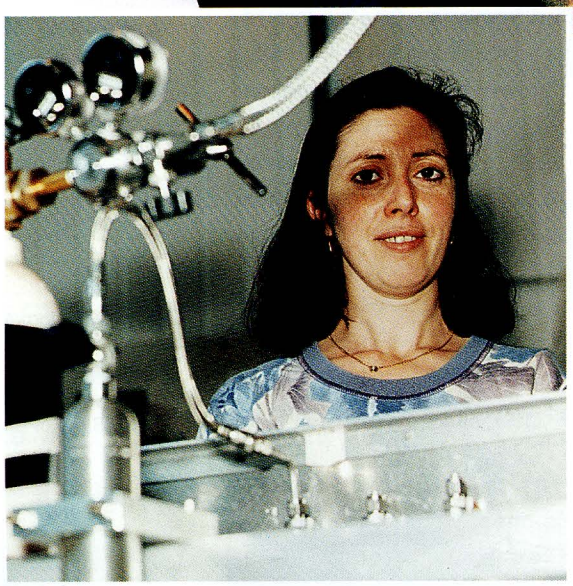
IRIS

On IRIS the data analysis software is indergoing considerable development, particularly a graphical interface for the IRIS Data Analysis package, IDA. The IRIS computer is now been upgraded to an Alpha Workstation.

Many different types of experiment were carried out on IRIS covering a broad range of scientific interest. This year quasielastic scattering studies have mainly concentrated on macromolecular systems including studies



Top left: Simon Carling and Ian Watts (Royal Institution, London) cooling an 'orange' cryostat to investigate magnetic oxalates on MuSR [97RC2493]. Top right: Dirk Visser (Warwick) and Andy Monteith (Royal Institution) using PRISMA to study phonons in $RbNiCl_3$ [97RC2463]. Centre: Robert Pugh, David Joyce and Grenville Jones (Salford) reflecting on their CrCuPt magnetic layer data from CRISP [97RC2640]. Lower left: Valeria Arrighi (Herriot-Watt) investigating polymers on IRIS [97RC2844]. Lower right: Yan Wang and Shulon Dong (UMIST) preparing to study single crystal ice on TFXA [97RC2635].



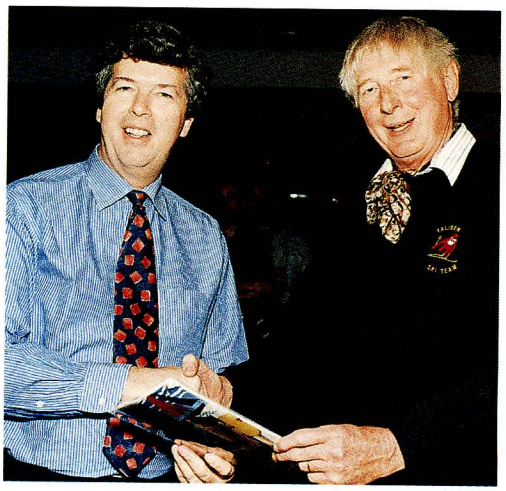
News and Events

Biology Support Laboratory

A national centre for high field NMR is to be built at RAL financed by a Joint Research Equipment Initiative grant awarded to Professor Tony Watts of Oxford University in collaboration with the Italian CNR. Part of the award, together with the matching Italian money, will go towards providing a biological support laboratory for ISIS.

Bruce Forsyth

Bruce Forsyth retired from RAL in March, having worked at RAL for 25 years. At his retirement presentation, Andrew Taylor spoke of Bruce's background at Cambridge and the RAF and his development as something of a polymath. His enthusiasm and energy were important to the then Neutron Beam Research Unit in pre-ISIS days. Promotion to an individual merit post soon followed and Bruce's career developed in instrument building both at RAL and the ILL, Grenoble, in science, and in computing. His computing skills were instrumental in getting the data acquisition system at ISIS off the ground in the early days and his acronym PuNCH (Pulsed Neutron Computer Hierarchy) lives on at ISIS today. His main scientific contribution has been in crystallography, principally magnetic structures, and specifically exploiting the scattering of polarised neutrons, in which area he has made many innovations in tandem with his long time collaborator Jane Brown of the ILL. As well as writing many papers on the subject, he has been instrumental in the team producing the CCSL (Cambridge Crystallographic Subroutine Library) suite of software which is widely used in the scientific community and is still a prominent feature in much of the crystallography performed at ISIS today.



Andrew Taylor and Bruce Forsyth on the occasion of Bruce's retirement [97RC1773].

Bruce will not be retiring altogether. He becomes an honorary Research Fellow and has a position at the Clarendon Laboratory. In addition, his Visiting Professorship at the University of Warwick continues, and he is heavily involved in a CRG developing X-ray Magnetic Scattering (XMaS - another of Bruce's acronyms!) at the ESRF in Grenoble.

Professor Tony Watts with his team from Oxford Biochemistry view the proposed site of the new NMR centre accompanied by Gordon Walker, Andrew Taylor, Colin Carlile and Brian Boland [96RC5477]

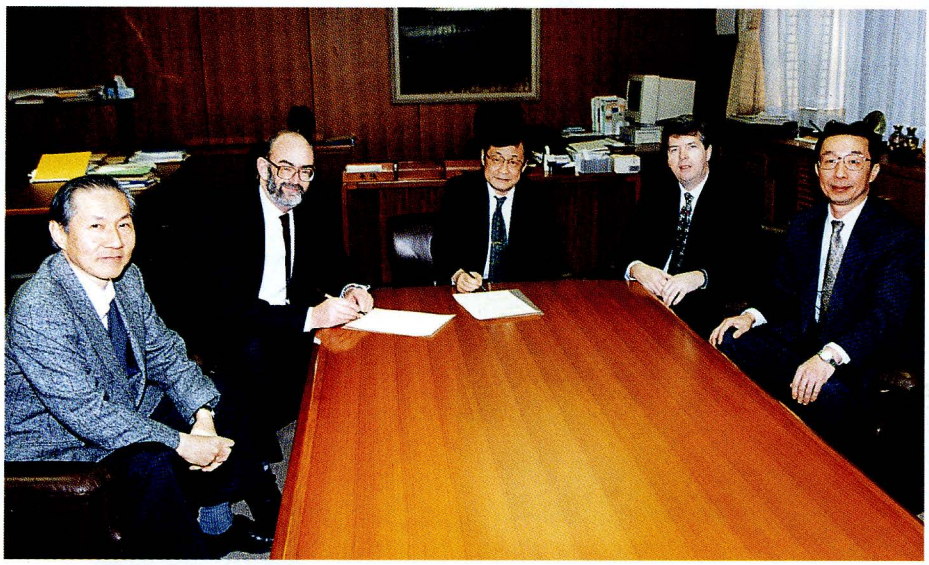
New Agreements - Japan

A new agreement beteen CCLRC and KEK was signed earlier this year, which extends the current collaboration for a further ten years.

NMBUM Meeting

The Neutron and Muon Beam Users meeting was held at RAL in September of last year under the auspicies of EPSRC. The next meeting will also be at RAL on 23 and 24 September 1997.

The Japan – ISIS Partnership



Above: Dr Kimura, Dr P R WIlliams, Professor H Sugawara (Director Gneral, KEK), Dr A Taylor and Professor H Ikeda. Dr Williams and Professor Sugawara are signing the CCLRC-KEK agreement.
Left: Dr. H. Kamitsubo, Head of the SPring-8 light source and Executive Director of RIKEN, and Dr Sakairi, SPring-8, tour ISIS with Mike Johnson (left), Katsu Ishida (second from right) and Gordon Eaton [97RC2092].
Below: A delegation from the Japanese parliament visiting ISIS [97RC3341].