# ANNUAL REPORT 88

INSTITUT MAX VON LAUE-PAUL LANGEVIN-GRENOBLE-FRANCE

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# Application for the use of ILL facilities

All research proposals have to be submitted to the Scientific Council for approval. The Council meets twice each year and the closing dates for the acceptance of applications are:

**February 15 and August 31.**

The completed research proposal forms should be sent to:

Scientific Coordination and Public Relations Office
(SCAPRO)
Institut Max von Laue - Paul Langevin
156X
38042 Grenoble Cedex
France
Tel. 76 48 72 44 B. Maier
76 48 71 79 H. Blank
76 48 70 41 K. Mayer-Jenkins (Secretary)
76 48 70 82 D. Dijoux (Secretary)
Telex: 320621 F

(Appropriate application forms may be obtained on request from the above office).

Under normal circumstances the ILL makes no charge for the use of its facilities. However, special equipment (other than the existing instruments, counters, standard cryostats and shielding requirements) must be provided by the user. This applies particularly to the experimental samples which must, in all cases, be provided by the user. Chemistry and Biology laboratory facilities are available for any necessary sample preparation.

The ILL makes a limited contribution towards the travel and subsistence expenses for experimentalists coming from approved laboratories in the five member countries (details on request).

## Commercially exploitable results

Visitors and ILL scientists may occasionally be involved in experiments which have possible commercial applications. If any scientists considers that this is the case, he should get in touch with the Scientific Secretary.

## Other publications available

Guide to Neutron Research Facilities, Edition 1988/89,
available from SCAPRO.

Experimental Reports and Theory College Activities 1988
available from the ILL Library.

## Front Cover

Fish-eye view of the ILL main building .

# Contents

The Institut Max von Laue - Paul Langevin

# The Institut Max von Laue - Paul Langevin

The Institut Max von Laue-Langevin (ILL) at Grenoble was formally founded in January 1967, with the signature of an intergovernmental convention between France and the Federal Republic of Germany. The aim was to provide the scientific community of the affiliated countries with a unique neutron beam facility applicable in fields such as the physics of condensed matter, chemistry, biology, nuclear physics and materials science. The construction of the Institute and its high flux reactor was undertaken as a joint French-German project, with a total capital investment of 335 million FF. The reactor went critical in August 1971 and reached its full power of 57 MW for the first time in December 1971. The year 1972 saw the start-up of the cold and hot sources, the first instruments and the beginning of the experimental programme.

On January 1st, 1973 the United Kingdom joined the Institute as a third equal partner, contributing its share to the total capital investment. The corresponding intergovernmental convention was formally signed in July 1974 by the pertinent ministers from the three affiliated countries. On December 9, 1981 a protocol was signed by representatives from the three member countries which extended the agreement until 1992 and beyond unless two years' notice of termination has been given.

In December 1986, an agreement on "Scientific Membership" of Spain was signed by the ILL and the Spanish Interministerial Commission on Science and Technology for a period of five years starting on January 1st, 1987. Switzerland became another "associated scientific member" of the Institute in 1988. The pertinent agreement between the ILL and the Swiss Bundesrat für Bildung und Wissenschaft was signed in May 1988.*

The ILL is a non-trading company under French civil law.

The three countries are represented by the following Associates:

- Kernforschungszentrum Karlsruhe GmbH, Germany
- Centre National de la Recherche Scientifique, France
- Commissariat à l'Energie Atomique, France
- Science and Engineering Research Council, United Kingdom.

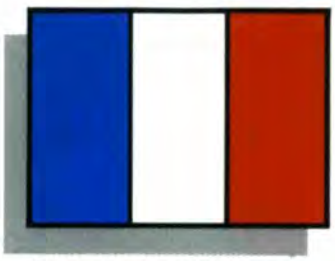
These Associates are represented on a Steering Committee, which establishes the general rules of the management of the ILL. The Institute is headed by a Director and two Assistant Directors, all with a five year tenure, the former to be nominated alternately by the German and the British Associates, the other two by the remaining Associates. A Scientific Council, nominated by the Associates, advises the Directors on the scientific programme and on practical aspects relating to its operation. The scientific users' community of the ILL is represented in 8 subcommittees of the Scientific Council, which meet twice a year to select those research proposals which are to be carried out at the neutron beam facilities of the ILL. A further subcommittee of the Scientific Council deals with questions of instrumentation, serving as a discussion platform between the ILL and its external users. The purpose of the ILL thus differs from other research Institutes in so far as it is a service Institute created so that chemistry, solid state physics, fundamental and nuclear physics, biology and metallurgy specialists from laboratories in the partner countries can use the unique power of neutron techniques to broaden the attack on their problems. Designing and operating instruments and helping the visiting users to carry out their experiments is thus the principal task of the Institute's own scientists. The experimental use of the instruments by ILL staff is subject to the same approval system as their use by external teams.

* Under these agreements Spanish and Swiss scientists have access to the ILL facilities under the same conditions as the three member countries and have the possibility of sending two thesis students. A seat is reserved for a Spanish and Swiss scientist in the ILL Scientific Council as well as membership in two of our sub-committees. The participation of Spain and Switzerland to the ILL Budget is limited to 1.5% each.

# External Organization of the ILL

## Associates of the ILL

Science and Engineering Research Council (SERC)



Commisariat à l'Energie Atomique (CEA)
Centre National de la Recherche Scientifique (CNRS)

Kernforschungszentrum Karlsruhe (KFK)

## Countries with scientific membership

Comisión Interministerial de Cienca y Tecnología (CICT)

Schweizer Bundesrat für Bildung und Wissenschaft (SBBW)

| **Steering Committee** (at its last meeting) | | |
|---|---|---|
| ■ Blaesing (BMFT)<br>■ Klose (KFT)<br>■ Peisl (Univ. Munich)<br>■ Schunck (BMFT) | ■ Comes (CNRS)<br>■ Cribier (CEA)<br>■ Fayard (CNRS)<br>■ Winter (CEA) | ■ Clark (SERC)<br>■ Enderby (Bristol)<br>■ Gray (RAL)<br>■ Newport (SERC) |

| **Scientific Council** | |
|---|---|
| **Plenary Session**<br>30 members | **Subcommittees**<br>68 members |

# General Organigram

## Directorate

Director:
W. Gläser

Co-Directors:
P. Day
A. Michaudon

## Departments

**Reactor Department**
Head: F. Franzetti
- Reactor Operation
- Operation of Sub-assemblies
- Site Security

**Instrument Operation Department**
Head: A. Heidemann
- Instrument Groups
- Central Group
- Sample Environment Group
- Cryogenics Service
- Site Maintenance

**Instrument and Methods Department**
Head: J.C. Faudou
- Instruments and Methods
- Construction & Mechanical Maintenance
- Electronics
- Monochromators
- Multi-detectors
- Special Instruments
- Multilayer Laboratory

**Computing Department**
Head: D. Rimmer
- Automation, Data Acquisition and Treatment
- Scientific Computing (DEC-1091)
- Telephone

**Administration Department**
Head: H.M. Spilker
- Personnel
- Finance & Purchasing
- Welfare & Staff Training
- Medical Assistance
- Translation Office
- Secretariat of the Steering Committee

## Directorate Services

**Scientific Coordination & Public Relations Office**
Head: B. Maier
- Co-ordination of the Experimental Programme
- Organisation of Workshops & Conferences
- Public Relations

**Délégué for Safety, Medical and Health Physics Group**
Bureau du Colombier

**Library**
Head: C. Castets

**Physicists' Secretariat**
Head: F. Parisot

## Scientific Sectors

**Colleges**
Convenors: College Secretaries
- **2** Theory
- **3** Fundamental and Nuclear Physics
- **4** Structural and Magnetic Excitations
- **5** Crystal and Magnetic Structures
- **6** Liquids, Disorder and Defects in Materials
- **8** Biology
- **9** Chemistry

**Physicists' Representatives**
Scientific Staff
- 66 Experimentalists
- 12 Theoreticians
- 30 Thesis students

**Instrument Groups**
Head: Senior Scientists
- Fundamental and Nuclear Physics Instruments
  Head: D. Dubbers
- Three-axis Spectrometers
  Head: B. Dorner
- TOF, High Resolution, SAS, Diffuse Scattering Instruments
  Heads: C. Janot, D. Richter
- Diffraction Instruments
  Head: J. Brown

31/12 1988

# Visits and Events in 1988


▲ *Signing of the official agreement on the scientific membership of Switzerland. On 13 May 1988 in the Chadwick amphitheatre.*
*The picture shows the signature of the document by U. Hochstrasser (Director of the Swiss Federal Office for Science and Education) and W. Gläser (ILL Director, right).*

◀ *On 22 February, 1988 M. Raymond Barre, MP and former French Prime Minister, visited the ILL.*

▲ *The French Minister for Research and Higher Education, M. Jacques Valade (centre), visited the ILL on 17 March 1988 in the context of the ESRF project. Pictured with R. Haensel, Director General of the ESRF.*

▲ *The Minister held a press conference in the ILL amphitheatre. From left to right : A. Michaudon (ILL Assistant Director), A. Carignon (Mayor of Grenoble and French Minister for the Environment), J. VALADE, M. J. P. Proust (Préfet de l'Isère), R. Haensel (Director General ESRF).*

▲ *ICNS 88 The Conference Banquet was held in the park of the Château de Vizille. This setting had been chosen to commemorate the 200th anniversary of the French Revolution which started at Vizille in 1788.*

▲ *Sir Ewen Fergusson, British Ambassador to France, visited the ILL on 25 October 1988. He received explanations about the ILL HFR from F. Franzetti.*

▲ *In the picture P. Day (British Assistant Director of the ILL, left) and A. Miller (Scientific Director of the ESRF, right) explain the mock-up of the synchrotron to the Ambassador.*

# Bericht des Direktors

1988 war für das ILL ein aufregendes Jahr, voller Überraschungen, aber schliesslich doch ein erfolgreiches Jahr. Wichtigste Aufgabe des Instituts ist es, den Reaktor sicher zu betreiben und seine Instrumente für eine möglichst effektive Nutzung zur Verfügung zu stellen.

## Reaktorbetrieb und Programm

Im vergangenen Jahr waren besondere Anstrengungen erforderlich, um die Versorgung des Reaktors mit Brennstoff zu sichern. Neben der Umstellung der Brennelementfabrikation von zwei auf einen Hersteller waren erhebliche Verzögerungen bei der Lieferung von Uran zu bewältigen. Die erfolgreiche Lösung aller dieser Probleme, die ohne eine kompetente und engagierte Mannschaft nicht möglich gewesen wäre, hat auch die Voraussetzungen für eine zuverlässige Fortsetzung des Betriebs in den nächsten Jahren geschaffen.

Trotz der erwähnten Schwierigkeiten konnten im Jahr 1988 5 Reaktorzyklen durchgeführt werden. Von den insgesamt eingereichten 1130 Experimentiervorschlägen mit einer gewünschten Strahlzeit von 9164 Tagen, konnten 807 Experimente mit einer Gesamtzeit von 4765 Tagen verwirklicht werden.

Auf dem Gebiet der Entwicklung und des Baus von Instrumenten lag der Schwerpunkt weiterhin bei der Instrumentierung der Neutronenleiter der horizontalen kalten Quelle. Von den 5 geplanten Instrumenten (4 programmierbare und 1 S-Experiment) wurde das Dreiachsenspektrometer IN14 fertiggestellt und in die Programmierung der nächsten Messzyklen einbezogen. Das nn̄-Experiment konnte mit der Datenerfassung beginnen. Das hochauflösende UCN-Spektrometer NESSIE ist in den Testbetrieb gegangen. Es ist geplant, das Rückstreuspektrometer IN10C, das Spinechospektrometer IN15 und die Kleinwinkelstreuanlage D22 Ende 1989, bzw. Anfang 1990 fertigzustellen. Es muss jedoch erwähnt werden, dass darüberhinaus die Verbesserung und Reparatur älterer Geräte einen wachsenden Arbeitsaufwand mit sich bringen.

Um die Spitzenposition des ILL zu halten und zu festigen, ist wieder ein umfassendes Modernisierungsprogramm erforderlich. Wissenschaftlicher Rat und Lenkungsausschuss haben empfohlen, ein solches Programm vorzubereiten.

## Wissenschaftliche Ergebnisse

Die 1988 durchgeführten wissenschaftlichen Arbeiten und erzielten Ergebnisse werden in den Berichten der Colleges ausführlicher beschrieben. Hier sollen nur einige Ergebnisse hervorgehoben werden.

Die bei der Kleinwinkelstreuung angewandte Kontrastvariationstechnik hat sich zu einem wichtigen Werkzeug zur Untersuchung komplexer biologischer Strukturen entwickelt. Wegen der grossen Streudichteunterschiede von Proteinen und Nukleinsäuren eignet sie sich besonders zur Untersuchung der Struktur von Komplexen aus beiden Molekültypen. Neben zahlreichen anderen Ergebnissen soll hier die erstmalige Bestimmung der lokalen Anordnung von Repressoren auf der DNA Doppelhelix erwähnt werden, die eine wichtige Rolle bei der Transkription spielen.

Mit der Kleinwinkelstreutechnik konnte auch ein Beitrag zum Verständnis der Rolle sterischer Lösungsmittelunterschiede bei der Aggregation von organischen Gelen geleistet werden, sicher ein wesentlicher Beitrag zum Verständnis der Eigenschaften von Kolloiden.

Bei den Untersuchungen der einfachen, ungeordneten oder teilgeordneten Systeme sind vor allem die Experimente zur Dynamik des Glasübergangs und zum Aufbau der quasikristallinen Systeme zu nennen. Ein erfolgversprechender methodischer Fortschritt wurde 1988 mit den ersten Testexperimenten zur Brillouinstreuung an Fluiden gemacht.

Aus dem Bereich der "klassischen" Neutronenkristallographie sind wohl vor allem die neuen Ergebnisse über die Ladungsdichtewellen in a-Uran und die magnetischen Eigenschaften von kubischen Laves-Phasen zu erwähnen. Die Pulverdiffraktometrie wurde erfolgreich zur Verfeinerung der Strukturen der neueren "high $T_c$" Bi- und Tl-Oxid-Materialien eingesetzt. Schliesslich sind die Fortschritte bei der zeitauflösenden Diffraktometrie zu nennen, die jetzt erlauben, periodische Prozesse im Mikrosekundenbereich zu studieren.

Das grosse Interesse an den "high $T_c$" Materialien hat auch zu zahlreichen spektroskopischen Untersuchungen geführt. Mit dem 3-Achsenspektrometer IN1 an der heissen Quelle steht ein einzigartiges Instrument für die Untersuchung hochenergetischer Anregungen zur Verfügung. Während im nichtsupraleitenden $La_2CuO_4$ hochenergetische Magnonen gefunden wurden, scheinen diese bei Dotierung der Probe mit Ba stark gedämpft zu werden. Mit polarisierten Neutronen und Polarisationsanalyse (IN20) konnten detaillierte Aussagen über Struktur und magnetische Anregungen in amorphen Ferromagneten gemacht werden.

Auch im Bereich der Kern- und Grundlagenphysik konnte eine Reihe schöner Ergebnisse erzielt werden. 50 Jahre nach der Entdeckung der Kernspaltung treten bei detaillierten Untersuchungen immer wieder Überraschungen auf, so wurde 1988 u.a. an PN1 (Lohengrin) bei der extrem asymmetrischen Spaltung von $^{235}U$, $^{239}Pu$ und $^{241}Am$ ein unerwarteter Neutronenpaarungseffekt entdeckt.

Mit der jetzigen verfügbaren intensiven Quelle ultrakalter Neutronen sind einige grundlegende Experimente zur Charakterisierung der Eigenschaften des Neutrons in Gang gekommen, wie z.B. die genauere Bestimmung der Neutronenlebensdauer. Die seit langem laufende Suche nach einem elektrischen Dipolmoment des Neutrons hat 1988 zum ersten Mal zu einem von null verschiedenen Wert geführt.

Neben dem eigentlichen experimentellen Programm hat das Institut eine Reihe von Konferenzen und Workshops organisiert oder sich an deren Organisation aktiv beteiligt. Hier sind zu nennen:

- die CODEST-Konferenz über quasikristalline Materialien,
- der Workshop über "Dynamics of Disordered Materials",
- die Internationale Konferenz über Neutronenstreuung (ICNS'88),

wobei insbesondere die ICNS'88 eine gute Gelegenheit war, die am ILL erzielten Forschungsergebnisse mit den Leistungen anderer Neutronenforschungszentren zu vergleichen.

## Zusammenarbeit mit ESRF

Das ILL hat auch 1988 das Mandat zur Vertretung der ESRF (European Synchrotron Radiation Facility) bis zu deren rechtsförmlichen Gründung wahrgenommen und die Gründungsmannschaft in jeder Hinsicht mit ihren Einrichtungen und Diensten unterstützt.

In der Vorbereitung der technischen und wissenschaftlichen Zusammenarbeit wurden im vergangenen Jahr grosse Fortschritte erzielt. Es wurde ein gemeinsames Management-Komitee gegründet, das regelmässig tagt und Angelegenheiten von gemeinsamem Interesse bespricht. Im Mai 1988 wurde von ILL und ESRF mit dem CEA ein Pachtvertrag für ein einziges gemeinsames Gelände unterzeichnet. Dieser gemeinsame Pachtvertrag wird durch einen internen Vertrag zwischen ILL und ESRF ergänzt, der den Rahmen für die Zusammenarbeit absteckt.

Als erste konkrete Massnahme wurde der Bau eines gemeinsamen Gebäudes beschlossen, der gemeinsame Kantine, Cafeteria, Bibliothek und Räumlichkeiten für die Theoriegruppen vorsieht. Weiter wurde eine Arbeitsgruppe ins Leben gerufen, die Vorbereitungen für die gemeinsame Geländeverwaltung und die Schaffung gemeinsamer Dienste treffen soll. Schliesslich ist vorgesehen, im kommenden Jahr mit der Veranstaltung gemeinsamer wissenschaftlicher Kolloquien und Workshops zu beginnen.

## Personalia

Im vergangenen Jahr hat es im Institut auch einige personelle Veränderungen gegeben. Ende August hat J. Enderby seine 3-jährige Tätigkeit als britischer Direktor beendet und ist an die Universität Bristol zurückgekehrt. Sein Nachfolger P. Day aus Oxford hat sein Amt am 1. Oktober angetreten. Nach 5-jähriger Tätigkeit am ILL ist auch der französische Direktor A. Michaudon zum CEA zurückgekehrt.

Weiter hat es auch einen Wechsel an der Spitze der Verwaltung gegeben. Chr. Eitner ist nach 5-jähriger Tätigkeit aus dem ILL ausgeschieden, seine Nachfolge hat H.-M. Spilker angetreten. Den ausgeschiedenen Herren sei an dieser Stelle noch einmal herzlich für ihre engagierte Mitarbeit zum Wohle des ILL gedankt.

Auch 1988 sind Mitarbeiter des Instituts ausgezeichnet worden oder haben Rufe erhalten. P. Nozières hat die Goldmedaille des CNRS erhalten. J. Pannetier hat einen Ruf auf eine Professur an der Universität Grenoble erhalten und D. Richter auf eine Institutsleiterstelle der KFA Jülich.

Wir betrachten diese Anerkennungen auch als Auszeichnungen des ganzen Instituts. Im Berichtszeitraum haben wieder eine Reihe bekannter Persönlichkeiten aus Politik und Wissenschaft das Institut besucht, u.a. sind zu nennen R. Barre (ehemaliger franz. Ministerpräsident), der britische Botschafter in Frankreich, Sir Ewen Fergusson, der Nobelpreisträger W.E. Lamb, der Direktor des Argonne National Laboratory A. Schriessheim,...

Schliesslich darf nicht unerwähnt bleiben, dass nach Spanien 1988 auch die Schweiz wissenschaftliches Mitglied des ILL geworden ist.

*Auch dieses Jahr obliegt mir die traurige Pflicht, vom Ableben eines langjährigen verdienten Mitarbeiters zu berichten. Monsieur P. Blum, als Ingenieur in der Abteilung "Instrumente und Methoden" seit Gründung des ILL tätig und u.a. massgebend an der Konstruktion von Neutronenleitern beteiligt, war uns allen in seinem Engagement für das ILL und seiner diskreten und zuvorkommenden Art ein Vorbild.*

*Wir werden ihn in besonderer Erinnerung behalten.*

W. Gläser

# Director's Report

1988 was a stimulating year for ILL, full of surprises but successful as a whole. The ILL's most important function is to operate the Reactor safely, and to make available its instruments for the most efficient utilization possible.

## Reactor Operation and Programme

Particular efforts were necessary during the year to ensure the supply of fuel for the Reactor. In addition to the change in fuel element manufacture from two suppliers to one, there were considerable delays in the supply of uranium. The successful solution of all these problems, which would have been impossible without a skilled and committed team, has also provided the conditions necessary for the reliable continuation of operation in the coming years.

Despite these difficulties, it was possible to operate five reactor cycles in 1988. Of the total of 1130 experiment proposals requesting beam-time of 9164 days, it was possible to carry out 807 experiments with a total beam-time of 4765 days.

In the development and construction of instruments, the emphasis continued to be on the instrumentation of the neutron guides for the Horizontal Cold Source. Of the five instruments planned (four scheduled and one 'S' instrument) the three-axis spectrometer IN14 was completed and included in the scheduling for the next cycles. The nn-experiment was able to start data acquisition. The high resolution UCN spectrometer NESSIE started test operation, and it is planned to complete the backscattering spectrometer IN10C, the spin-echo spectrometer IN15 and the small-angle scattering instrument D22 at the end of 1989 or early in 1990. It should however be mentioned that the improvement and repair of older instruments also results in an increasing workload.

To maintain and strengthen the ILL's leading position, an extensive Modernisation Programme is again necessary. The Scientific Council and Steering Committee have recommended the preparation of such a programme.

## Scientific Results

The scientific work carried out in 1988 and the results achieved will be described in more detail in the college reports. Here attention is drawn to a few particular results.

The contrast variation technique used in small-angle scattering has developed into an important tool for the study of complex biological structures. Because of the considerable differences in scattering length density of proteins and nucleic acids this is particularly suitable for the study of the structure of complexes containing both types of molecule. In addition to numerous other results, mention should be made of the first determination of the arrangement on the DNA double helix of repressors, which play an important part in transcription.

Small-angle scattering also made a contribution to the understanding of the role of steric solvent differences in the aggregation of organic gels, certainly an important contribution to the understanding of the properties of colloids.

In the studies of simple disordered or partially ordered systems, reference should be made in particular to the experiments on dynamics of the glass transition and the construction of quasi-crystalline systems. A promising advance as regards methods was made in 1988 with the first test experiments on Brillouin scattering on fluids.

In the field of 'classical' neutron crystallography, particular mention should be made of the new results on charge-density waves in a-Uranium and the magnetic properties of cubic Laves phases. Powder diffractometry was successfully used to refine the structures of the new high $T_c$ Bi and Tl oxide materials. Finally progress has been made in time resolved diffractometry, which now permits the study of periodic processes in the microsecond range.

The great interest in high $T_c$ materials has also led to numerous spectroscopic studies. The three-axis spectrometer IN1 on the hot source is a unique instrument for the study of high energy excitations. Whereas high-energy magnons were found in non-superconducting $La_2CuO_4$, these appear to be considerably attenuated when the sample is doped with Ba. With polarised neutrons and polarisation analysis (IN20) detailed information was found on structure and magnetic excitations in amorphous ferromagnets.

A number of excellent results were also obtained in nuclear and fundamental physics. Fifty years after the discovery of nuclear fission surprising discoveries are still being made in detailed studies; for example in 1988 on PN1 (Lohengrin) an unexpected neutron pairing effect was discovered in extremely asymmetrical fission of $^{235}U$, $^{239}Pu$ and $^{241}Am$.

With the intense ultra-cold neutron source now available some fundamental experiments to characterise the properties of the neutron have started, for example the exact determination of the neutron lifetime. In 1988 for the first time the long-running search for a neutron electric dipole moment led to a value other than zero.

In addition to the actual experimental programme the ILL organised a number of conferences and workshops, or participated actively in their organisation. Particular mention should be made of:

- the CODEST conference on quasi-crystalline materials,
- the workshop on the dynamics of disordered materials,
- the international conference on neutron scattering (ICNS'88).

In particular the ICNS'88 was a good opportunity to compare the research results obtained at ILL with those of other neutron research centres.

## Collaboration with the ESRF

In 1988 the ILL continued to act on behalf of the ESRF (European Synchrotron Radiation Facility) until its formal foundation, and supported the project team in all possible ways with its facilities and services.

Great progress was achieved during the year in the preparations for technical and scientific collaboration. A joint Management Committee was set up, which meets regularly and discusses matters of common interest. In May 1988 the ILL and ESRF signed a lease contract with the CEA for a single common site. An internal contract between ILL and ESRF defining the framework for the collaboration complements this joint lease contract.

As a first concrete measure it was decided to build a common building incorporating a joint canteen, cafeteria, library and offices for the theory groups. A working group was also set up, to make preparations for the Joint Site Administration and the establishment of common services. Finally it is intended to start organising joint scientific colloquia and workshops in the coming year.

## Personnel

There were also some changes in personnel at the ILL during the year. At the end of August J. Enderby completed his three year term as British Director, and returned to the University of Bristol. His successor, P. Day from Oxford took up his post on 1 October. The French Director A. Michaudon also returned to the CEA after five years at the ILL.

There was also a change of Head of Administration. C. Eitner left after five years' work at ILL, and was succeeded by H. M. Spilker. I should like to express my sincere thanks to those who have left ILL for their commitment and for all they have done for the benefit of the Institute.

In 1988 ILL staff members were again awarded honours or appointments. P. Nozières received the Gold Medal of the CNRS. J. Pannetier was appointed Professor at the University of Grenoble and D. Richter Department Head at the KFA Jülich.

We consider these distinctions as an honour for the ILL as a whole. During the year a number of well-known political and scientific personalities again visited the ILL, including R. Barre (former French Prime Minister), the British Ambassador in France, Sir Ewen Fergusson, the Nobel Prize winner W.E. Lamb and the Director of the Argonne National Laboratory, A. Schriessheim.

It should be mentioned that in 1988 Switzerland followed Spain in taking up scientific membership of the ILL.

*This year again I have the sad task of recording the death of an employee who had worked for many years at the ILL. Paul Blum, an engineer in the Instruments and Methods Department, had been at ILL since its foundation; his work included a major contribution to the design of neutron guides. He will be remembered for his commitment to the ILL and for his discretion and helpfulness.*

W. Gläser

# Rapport du Directeur

L'année 1988 a été une bonne année pour l'ILL, certes pleine de surprises, mais fructueuse dans l'ensemble. La tâche principale de l'ILL est, comme chacun sait, d'assurer le fonctionnement du réacteur en toute sécurité et de fournir des instruments d'expérience efficaces.

## Le Réacteur et les Programmes d'Expériences

Pendant l'année écoulée, l'approvisionnement du réacteur en combustible ne s'est pas fait sans difficultés. Il a fallu faire face à des retards de livraison considérables, et, de plus, la mise en forme des éléments combustibles a dû être réservée à un seul des deux fabricants habituels. Des solutions satisfaisantes ont finalement été trouvées pour tous ces problèmes, grâce à la compétence et à l'engagement de l'équipe responsable. Les conditions de confiance en un bon fonctionnement au cours des années à venir ont ainsi été créées.

Il a été malgré tout possible de faire fonctionner le réacteur pendant 5 cycles. En conséquence, 807 expériences correspondant à un temps de faisceaux total de 4765 jours ont pu être réalisées sur un total de 1130 propositions et 9164 jours de temps de faisceaux demandés.

Les développements et constructions d'instruments ont essentiellement eu pour but d'équiper les guides de neutrons récemment installés sur la source froide horizontale. Cinq instruments doivent ainsi venir compléter les possibilités d'expériences : quatre instruments entrant dans la planification habituelle et un instrument dit "S". Le spectromètre à trois axes IN14 a été achevé et sera inclus dans les planifications d'expérience des prochains cycles. Les appareils d'acquisition de données pour l'expérience $n\bar{n}$ sont maintenant au point. Le spectromètre à haute résolution NESSIE, utilisant des neutrons ultra-froids, en est à sa période d'essais et il est prévu d'achever la construction du spectromètre à rétrodiffusion IN10C, du spectromètre à écho de spin IN15 et de l'appareil de diffusion aux petits angles D22 vers la fin de 1989 ou au début de 1990. Il faut finalement souligner que de plus en plus d'efforts doivent être fournis pour maintenir en bon état de marche les instruments datant des débuts de l'ILL. Il semble donc raisonnable d'envisager dès maintenant un important programme de modernisation si l'on veut garder à l'ILL son "leadership" en recherche neutronique. Le Conseil Scientifique et le Comité de Direction ont, de fait, recommandé la préparation d'un tel programme.

## Les Résultats Scientifiques

Le détail des résultats obtenus cette année est repris, comme d'habitude, dans les rapports des Secrétaires de Collèges. Seuls quelques points particuliers seront soulignés ici.

Des structures biologiques complexes ont pu être étudiées grâce à la méthode de variation de contraste en diffusion aux petits angles. En effet, les densités de longueur de diffusion varient beaucoup lorsqu'on passe des protéines aux acides nucléiques et il est ainsi possible d'analyser les corrélations structurales dans des édifices compliqués où les deux types de molécules coexistent.

On peut citer en particulier la première détermination de l'arrangement local des répresseurs dans la double hélice d'ADN, dont le rôle est fondamental pour les mécanismes de transcription héréditaires.

La diffusion aux petits angles a aussi permis de comprendre les effets stériques de solvants dans les processus de formation de gels, ce qui est certainement une étape importante vers une meilleure connaissance des propriétés des colloïdes.

Les études consacrées aux systèmes non-cristallins les plus simples ont conduit à des résultats très intéressants. En particulier, la dynamique de transitions vitreuses et une description de systèmes quasi-cristallins ont fait l'objet d'expériences réussies. Des essais prometteurs sur des fluides simples ont également montré que des mesures de diffusion Brillouin pouvaient raisonnablement être envisagées.

La cristallographie "classique" a aussi eu ses succès avec, notamment, la mise en évidence d'ondes de densité de charge dans l'uranium a et des propriétés magnétiques de phases de Laves cubiques. La diffractométrie de poudre s'est spécialement distinguée dans les raffinements de structures des nouveaux oxydes supraconducteurs à haute $T_c$, à base de Bi et Tl. Enfin, la diffractométrie en temps réel a maintenant atteint une telle qualité qu'il est possible de suivre des processus périodiques dans le domaine de la micro-seconde.

Les matériaux supraconducteurs à haute $T_c$ ont également initié un très grand nombre d'études spectroscopiques. Le spectromètre 3-axes IN1 et son faisceau de neutrons chauds est un instrument tout à fait exceptionnel pour l'approche des excitations de haute énergie. Ainsi les magnons qui se manifestent dans le $La_2CuO_4$ non-supraconducteur semblent fortement atténués par le dopage au Ba. D'autres matériaux non classiques comme certains alliages amorphes ferromagnétiques ont bénéficié d'investigations en neutrons polarisés et analyse de polarisation (IN20) pour préciser leur structure et la nature des excitations magnétiques qui s'y manifestent.

La physique nucléaire et fondamentale n'est pas en reste dans le palmarès de ces résultats d'exception. 50 ans après la première observation du phénomène de fission nucléaire, on faisait encore de surprenantes découvertes dans ce domaine. Par exemple, des fissions très dissymétriques de $^{235}U$, $^{239}Pu$ et $^{241}Am$ ont mis en évidence un effet inattendu d'appariement de neutrons (PN1 Lohengrin).

Finalement, il faut encore citer la possibilité nouvelle d'utiliser les faisceaux intenses de neutrons ultra-froids, pour étudier les propriétés du neutron lui-même, comme par exemple sa durée de vie ou son moment dipolaire électrique pour lequel une valeur non nulle a été mesurée pour la première fois en 1988.

En complément de son programme d'activités expérimentales, l'ILL a aussi organisé ou activement participé à différentes manifestations scientifiques comme

- la conférence ILL/CODEST sur les quasi-cristaux,
- le "workshop" sur la dynamique des matériaux désordonnés,
- et la Conférence Internationale sur la Diffraction des Neutrons (ICNS'88).

Cette dernière, notamment, fut une bonne occasion de confronter les travaux de l'ILL à ceux des autres centres de recherche neutronique.

## Collaboration avec l'ESRF

En 1988, l'ILL a continué à représenter légalement l'ESRF (Source Européenne de Rayonnement Synchrotron) jusqu'à sa mise en place officielle. L'équipe chargée du projet a pu bénéficier pleinement de l'aide des services et installations de l'ILL.

Des progrès importants ont été réalisés en 1988 en ce qui concerne la préparation de la collaboration technique et scientifique. Un comité commun de liaison au niveau des directions a été créé, il se réunit régulièrement pour discuter des questions d'intérêt commun. En mai 1988, l'ILL et l'ESRF ont signé conjointement un contrat de bail avec le CEA en vue de la création d'un site commun. Ce contrat de bail est complété par un contrat interne entre l'ILL et l'ESRF qui fixe le cadre de la collaboration.

En tant que première mesure concrète, il a été décidé de construire un bâtiment commun, dans lequel sont prévus une cantine, une cafétéria, une bibliothèque et des bureaux pour les groupes théorie des deux instituts. D'autre part, un groupe de travail a été constitué, qui est chargé d'effectuer les préparatifs en vue de l'administration commune du site et de la création de services communs aux deux instituts.

Finalement, il est prévu d'entreprendre au cours de l'année prochaine l'organisation de colloques et de "workshops" communs.

## Questions de Personnel

Quelques changements ont eu lieu à l'Institut au cours de l'année écoulée. Le mandat de 3 ans de J. Enderby, en tant que directeur britannique, s'est terminé fin août, M. Enderby est retourné à l'université de Bristol. M. Day, d'Oxford, lui a succédé le 1er octobre. M. Michaudon, le directeur français, est retourné au CEA après un mandat de 5 ans à l'ILL.

Un changement a également eu lieu à la tête de l'Administration. Chr. Eitner a quitté l'ILL après 5 ans d'activité, H. M. Spilker lui a succédé. Je voudrais une fois encore exprimer mes sincères remerciements à ces personnes pour leur collaboration et leur engagement au service de l'ILL.

En 1988 également, des membres de l'Institut ont reçu des prix et des distinctions. P. Nozières a reçu la médaille d'or du CNRS. J. Pannetier fut nommé Professeur à l'université de Grenoble et D. Richter Chef de Departement au KFA Jülich.

Nous considérons ces distinctions comme un grand honneur pour l'Institut tout entier. Cette année encore plusieurs personnalités du monde politique et scientifique ont visité l'ILL, citons entre autres R. Barre (ancien Premier Ministre de la République Française), l'Ambassadeur Britannique en France, Sir Ewen Fergusson, le Prix Nobel W.E. Lamb, le Directeur d'Argonne National Laboratory, A. Schriessheim.

Enfin, il ne faut pas oublier de mentionner la participation scientifique de la Suisse, qui après celle de l'Espagne, est venue enrichir le potentiel de l'ILL.

*Cette année encore j'ai le triste devoir de vous faire part du décès d'un collaborateur qui a travaillé à l'ILL pendant de nombreuses années. Monsieur P. Blum, ingénieur dans le département "Instruments et Méthodes", était à l'ILL depuis sa fondation. Il a, en particulier, participé de manière active à la construction des guides de neutrons. Nous garderons un excellent souvenir de sa présence efficace et discrète et de son engagement au service de l'ILL.*

W. Gläser

# Collaboration ILL/ESRF

Several years have passed since the ILL in 1984 made the proposal to build the European Synchrotron Radiation Facility (ESRF) next to its site. This proposal became a reality on December 16, 1988 with the signature of the official intergovernmental agreement creating the ESRF, located in Grenoble near the ILL. The main argument put forward by the ILL in its proposal was the overall synergy between two Institutes centered on neutron and photon sources, known to be complementary probes for the study of condensed matter.

A close cooperation and a fruitful cross-fertilization on the scientific level should result, when the ESRF is in operation, in a scientific complex, international in character, of unmatched power and quality. But many practical advantages can also result at technical and administrative levels by sharing common services.

The ILL and the ESRF have not waited passively for the foundation of the ESRF to start working together and paving the way towards a closer cooperation in the years to come.

The first practical evidence of collaboration between the two Institutes was the erection in 1986 of a two-story building on the ILL site, funded by the ESRF and used to house the ESRF team. Since then and until its legal existence, the ESRF has used the ILL as trustee for many administrative actions. The ESRF also has access to many administrative, technical, scientific and general services of the ILL.

In order to examine and monitor all aspects of the collaboration between the two Institutes, a Joint Committee at Directorate level has been set up which meets at approximately monthly intervals.

A major step forward towards collaboration was the signature in May 1988, between the Commissariat à l'Energie Atomique and the two Institutes, of a lease for a common site. This lease identifies two areas reserved for the ILL and the ESRF as well as a small zone for common use. Exceptions are possible subject to the formal agreement of the three parties concerned (CEA, ILL, ESRF). In addition to the lease, an internal convention has been drawn up between the ILL and the ESRF to provide a framework for the common management of the site in order to make the best possible use of it. The part of the common site reserved for ESRF will be cleared by January 1989.

A joint site administration, headed by a member of the ESRF staff, will be set up shortly after the recommendations of a working group have been made.

Another major step forward was the proposal, endorsed in principle by the ILL Steering Committee and the ESRF Council to erect a joint building to house a canteen, a cafeteria, a library and a theory group for both institutes. This building is planned to have a useful area of 2540 $m^2$ and to be located on the ESRF zone in close proximity of the ILL. This operation has been approved because it is believed that it is an essential informal manner and facilitates communication and interaction between them. This project is now under study by the ESRF architect. Its cost, estimated at 18.7 MF, will be financed 2/3 by the ESRF and 1/3 by the ILL and its construction should take about three years, starting in 1989.

The personnel policies are also being studied together by the ILL and the ESRF and joint proposals are being made to the ESRF Council and the ILL Steering Committee.

A close collaboration is anticipated in the combined Theory Group which will be composed of theorists recruited separately by the two institutes in close association. In 1989, the ESRF has no posts for theorists but the ILL will use one of its posts to recruit a theorist proposed by the ESRF.

The ESRF is represented by an observer at the ILL Scientific Council and vice-versa

The combined scientific life will start in 1989 with the organisation of joint colloquia and of two joint workshops.

Several other areas of collaboration will be studied and implemented in 1989 and reported in the next Annual Report.

A. Michaudon

*The mock-up shows the ILL and the ESRF on the peninsula formed by the two rivers Drac and Isère.*

# College 2

# Theory

### Members of the College

C. Balseiro
H. Capellmann
D. Grempel
J. Jolie
A. Maggs
J. Rodriguez
T. Sluckin
P. Thalmeier
R. Bowley
P. Grangé
P. Holdsworth
M. Lavagna
P. Nozières
N. Schopohl
J. Solyom
J. Vannimenus

## Introduction

The scientific activities within the Theory College cover condensed matter physics and nuclear physics. In accordance with the composition of the college the majority belongs to the condensed matter area. Here the topics include superconductivity (with particular emphasis on the high $T_c$ ceramic compounds), liquids and liquid crystals, heavy fermions, disorder problems and localization, properties of helium and various other subjects (like low-dimensional systems, membranes, films, magnetic chains). The activities in nuclear physics include parity violation, extensions beyond mean field theory, 3-body forces, nuclear structure and the analysis of Gamma-Ray Induced Doppler broadening measurements carried out at the ILL.

## Scientific Trends and Highlights in 1988

### High Tc ceramic superconductors and related compounds

The activities are not only restricted to properties of the superconductors themselves, but are also directed towards the related compounds, similar in composition but not superconducting, which show a variety of highly interesting and unusual phenomena. Only a broad and general understanding of this class of systems can eventually lead to an explanation of the superconductivity, which of course remains the most important aspect.

There is rather convincing evidence and general agreement that the $CuO_2$ planes are the essential units for superconductivity, therefore the dominant effort is made to understand their properties. C. Balseiro is studying the electronic properties of these $CuO_2$ planes, focussing on Cu 3d and oxygen 2p orbitals and retaining both intra- and interatomic correlations. Both approximative methods on infinite systems as well as exact diagonalizations of finite clusters are used and compared to derive the metal-insulator and magnetic phase diagrams and to study elementary excitations. The results obtained can account for various features in photoemission spectra.

A considerable effort is made to understand the highly peculiar magnetic properties associated with the $CuO_2$ planes. Long-range magnetic order is observed in non-superconducting compositions (e.g. $La_2CuO_4$, $YBa_2Cu_3O_6$) with unusual quasi two-dimensional excitations remaining very sharp in q into the paramagnetic phase. The excitations have been widely interpreted as resulting from strong quasi two-dimensional short-range order (with correlation lengths $\xi$ of several hundred Å well above $T_N$). D. Grempel showed that this is a misleading concept because the true signature of "giant short-range order", which would be a strong quasi-elastic peak of narrow width in q **and** $\omega$, is missing. Grempel studied the dynamics of two dimensional quantum antiferromagnetics: for $q\xi << 1$ very slow dynamics are obtained for low temperatures (the "quasi-elastic peak"), whereas for $q\xi >> 1$ hydrodynamics breaks down but dynamic scaling remains valid, yielding spin-wave damping proportional to $\omega$. The interesting experimental properties (sharp excitations **without** a quasi-elastic peak) remain an open problem.

J. Rodriguez is studying the topological excitations in quantum 2d antiferromagnets, skyrmions, which can be thought of as domain wall loops in the 2-d antiferromagnet. Classically such defects can have any size and do not interact. Rodriguez showed that quantum corrections tend to expand the skyrmion and induce a repulsive skyrmion-skyrmion interaction.

H. Capellmann is collaborating with O. Schärpf, T. Brückel, and K. U. Neumann in an extensive experimental neutron scattering effort to explore the magnetic properties of high $T_c$ related materials. After establishing the absence of magnetic fluctuations in the thermal energy range in superconducting $YBa_2Cu_3O_7$ the studies were extended to lower oxygen concentrations, where superconductivity disappears and magnetic order and paramagnetic fluctuations are observed. These results are important for theoretical concepts of superconductivity (superconductivity apparently is in competition with magnetism not being caused by magnetic fluctuations).

The experimental studies are also extended to $RBa_2Cu_2O_{7-\delta}$ (R = rare earth elements), both for superconducting and nonsuperconducting compositions. Although the rare earth magnetic moments have essentially no effect on superconductivity, their ordered phases at very low temperatures and crystalline electric field transitions represent interesting problems.

P. Thalmeier developed a microscopic model to describe the antiferromagnetic structure of $ErBa_2Cu_3O_7$, with moments tilted against the **c**-axis.

J. Vannimenus is interested in structural phase transitions occurring in the superconducting cuprates, in particular oxygen vacancy ordering. Numerical model calculations are performed both at the ILL and on specialized computers.

M. Lavagna is studying the problem of electronic correlations in the Hubbard model. She developed a new approach which consists of introducing fluctuations around the Gutzwiller approximation.

## Intermediate valence and heavy fermions

The theoretical concept developed by H. Capellmann (in collaboration with K. U. Neumann and S. Lipinski) is based on the mutual coupling between magnetic, lattice, and slow charge fluctuations. It was shown that slow charge fluctuations can cause resistivity anomalies and magnetic transitions of f-moments. The experimental neutron scattering collaboration with K. U. Neumann, O. Schärpf, and K. Ziebeck continues. The aim is the study of the mutual influence of magnetic and lattice degrees of freedom.

P. Thalmeier is studying elastic anomalies in heavy fermion systems and Kondo alloys. Temperature and field dependence of elastic constants yield important information on electronic states and their coupling to the lattice. This has been discussed within the context of phenomenological as well as microscopic theories.

M. Lavagna discussed the onset of metamagnetism under applied magnetic fields. She explored whether the Anderson lattice model could give rise to such a prediction, the study was done within the Gutzwiller approximation.

## Disordered systems, localization

J. Vannimenus reanalyzed and extended various analytical and numerical results for frustrated vector spins (in collaboration with D. Haldane, C. Jayaprakash, and S. Kirkpatrick). The effects of antiferromagnetically coupling impurities in a ferromagnetic lattice were explored, yielding thresholds for the appearance of a canted ground state or the disappearance of long-range order at the expense of disordered states.

J. Vannimenus also studied the morphology of ramified patterns. Complex patterns with a distinctive morphology are observed in various domains (growth of aggregates, dendritic instabilities, viscous flows...). In collaboration with X. Viennot an approach to characterize the patterns was developed which is based on combinatorial methods originating in hydrogeology and computer science.

Among the important aspects of disordered systems an important role is provided by the statistics of the energy level distribution. In particular fluctuations in the number of levels contained in a given interval of energy and the rigidity of level repulsion effects are closely related to conductance fluctuations of a sample and to its response at low frequency.

D. Grempel studied the crossover between two extreme types of behaviour, Wigner statistics and Poisson statistics, as a function of size and disorder in two cases:

a) Anderson's model of a disordered alloy and
b) the periodically driven rotator.

P. Holdsworth investigated the effects of pairwise interactions which are non-separable in the molecular orientation and the vector joining the two particles in amorphous solid systems. He finds orientational glass-like behaviour. He also worked on phonon-assisted variable-range hopping conductivity in a system of localized electronic states. Calculations were performed for conductivity, thermopower, and thermal conductivity.

## Helium and related topics

R. Bowley, F. Graner, and P. Nozières collaborated in an investigation of the mobility of the liquid solid interface in $^3$He. The effective growth of $^3$He crystals has recently been measured near the minimum of the melting curve. The latent heat is essentially released on the liquid side and has to cross the Kapitza resistance of the interface to be evacuated via the solid. The thermodynamics of these processes were clarified. The same collaboration also investigated the effects of the interface mobility on the Mullins Sekerka instability and on stable growth shapes of crystals when facets are present.

R. Bowley studied the rate of vortex nucleation by negative ions in liquid helium. Recent measurements suggest that a loop of vortex is created, either tunneling through a potential barrier or being excited over it. Similar ideas are applied to granular films of high $T_c$ superconductors, where intergranular vortices can be created in pairs in the presence of an applied current.

N. Schopohl (in collaboration with D. Waxman) continued studies of the A-B phase boundary of superfluid $^3$He. Transmission and Andreev reflection of ballistic wave packets were calculated.

Together with G. Eilenberger, N. Schopohl studied the general representation of Cooper-pairing states with p-wave symmetry. It was shown that an explicit factorization and separation of the gauge and orientational degrees of freedom from the other degrees of freedom of the order parameter can be obtained. N. Schopohl and A. Baratoff investigated magnetic properties of extreme type II superconductors. Field distribution around a vortex line, the lower critical field and the vortex lattice associated with an anisotropic London tensor were computed.

## Liquids and liquid crystals

P. Nozières worked on a macroscopic description of the flow of suspensions, in analogy to the physics of dielectrics. In particular the averaging process is very delicate and has to be formulated very precisely.

Most of T. Sluckin's activity was concentrated on liquid crystals, in particular liquid crystal surfaces. The effects of a surface in inducing smectic order were studied, extensions being made to investigate the rich phase diagram of a Nematic-Isotropic-Smectic A system in finite geometry. Sluckin further studied polydisperse fluids to understand the blurring of the onset of liquid-crystal behaviour. N. Schopohl and T. Sluckin collaborated in an investigation of the core structure in nematic defects and its analogy to magnetic defects. Finally T. Sluckin continued work on adsorbed systems in the hope of understanding the nitrogen-argon mixture adsorbed on graphite.

P. Holdsworth's main interest has been in the study of correlations and interactions in two-dimensional liquid crystal systems. He studied the effects of pairwise interactions, both separable or non separable in the molecular orientations and the vector joining the two particles, using Monte Carlo simulation. Separable potentials yield quasi long-range order of molecular orientations in accord with Kosterlitz Thouless theory, but only short-range order of bond orientations.

# College 2

In contrast for non-separable potentials an anisotropic liquid phase is obtained with order in both the molecular and bond orientations.

## Other activities in condensed matter

A. Maggs is studying the statistical mechanics of amphiphilic membranes and films. He is trying to understand the entropic or steric interactions in membrane systems with the help of computer simulations. As a first step the entropic forces in rigid polymers are studied. The work is extended to the case of the full 2-d membrane. A variation on the case of lamellar membranes is provided by the case of "vesicles", closed quasi-spherical amphiphilic surfaces. From the theoretical point of view there is a new scaling variable to be considered beyond those in lamellar membranes: the pressure difference inside and outside the vesicle. Computer studies are made to study scaling and cross-over arguments.

T. Solyom in collaboration with J. Timonen, continued the study of one-dimensional composite spin models. Numerical results on finite chains are obtained to test predictions from field-theoretical analogies. Discrepancies are established possibly arising from strong renormalization effects which are not under control, as the field-theoretical continuum limit is taken.

Solyom also studied the possibility of exciton formation in rare-earth compounds and their consequence on photoemission spectra. Finally R. Bowley studied NMR imaging: The ISIS technique has been extended to allow one to select images of arbitrary shape rather than a simple cube. This will improve the resolution in studies of energy metabolism by phosphorus NMR spectroscopy.

## Nuclear physics

J. Jolie worked mainly on the analysis of the Gamma-Ray Induced Doppler broadening (GRID) measurements performed with the two-axis flat crystal spectrometer GAMS-4 and he continued his work in theoretical nuclear structure. Both activities were carried out in close collaboration with College 3.

Concerning GRID measurements and their theoretical interpretation, a previous description was improved with the use of the Born-Meyer potential and the introduction of a Maxwellian stopping peak. Good results were obtained for $^{49}$Ti, $^{54}$Cr, and $^{57}$Fe. For very short lifetimes the influence of gamma-gamma correlations were studied. Jolie's work in nuclear structure focussed on odd-odd nuclei, nuclei near closed shells, and odd-A nuclei.

P. Grangé studied extensions beyond the nuclear mean field description. Through dispersion relations between real and imaginary parts of the mass operator a constraining self consistency link exists between gain and loss terms and the nuclear mean field present in nuclear transport equations currently used in the description of energetic heavy ion collisions. Some arguments are also given that the corrections to the nuclear mean field go beyond the non-relativistic framework.

Grangé showed that the study of the optical model potential in a local and thermal approximation in the nuclear medium gives a consistent link between the nuclear mean field and residual two-body collisions in the Landau-Vlassov transport theory. He also studied parity violation asymmetry in nucleon-nucleon scattering, evaluating the helicity dependence of the cross-section for scattering longitudinally polarized neutrons on unpolarized neutrons. Finally in the nuclear many body problem Grangé established novel consistency requirements between the initial two body nucleon-nucleon force and the residual three body force.

Secretary: H. Capellmann

# Nuclear and Fundamental Physics

### Members of the College

**Internal members**

J. P. Bocquet
H. G. Börner
J. Copnell
W. Drexel
D. Dubbers
H. R Faust
I. Förster
P. Geltenbort
S. Judge
B. Krusche
W. Lippert
W. Mampe
R. A. Oliver
D. Richardson
S. J. Robinson
P. Schillebeeckx
K. Schreckenbach
J. L. Sida
A. Williams

**Long-Term Visitors**

W. Andrejtscheff (Belgrade)
G. Azuelos (Triumph)
F. Baumann (Bayreuth)
T. Bitter (Heidelberg)
H. Borel (Grenoble)
R. Brissot (CEN Grenoble)
J. Byrne (Sussex)
M. S. Dewey (NIST-USA)
K. Eder (Vienna)
R. Gähler (TUM)
R. Golub (HMI)
G. Greene (NIST-USA)
P. Grivot (ISN)
M. Grüber (Vienna)
E. Gutsmiedl (TUM)
W. D. Hamilton (Sussex)
E. Kessler (NIST-USA)
I. A. Kilvington (RAL)
R. Kossakowski (Annecy)
E. Krüger (PTB)
S. Lamoreaux (Seattle)
P. Liaud (Chambery)
W. Nistler (PTB)
M. Pendlebury (Sussex)
P. Petkov (Belgrade)
A. Steyrl (TUM)
D. Thompson (RAL)
P. Vittoz (Grenoble)
G. Vivier (Grenoble)
W. Weirauch (PTB)
A. Zeilinger (Vienna)

### Collaborations of College 3 with Institutes and Universities in 1988

Alger
Annecy (LAPP)
Argonne Nat. Lab.
Bayreuth
Belgrade
Berlin HMI
Bordeaux CEN
Braunschweig PTB
Braunschweig Univ.
Bruyères-Le-Chatel
Chambery
Darmstadt GSI
Darmstadt TU
Fribourg Univ.
Gaithersburg NIST
Geel JRC/CBNM
Gent Univ.
Göttingen Univ.
Grenoble CEN
Grenoble ISN
Grenoble Univ.
Harvard Univ.
Heidelberg Univ.
Karlsruhe KFA
Karlsruhe Univ.
Köln Univ.
Livermore LLNL
London, Imperial College
Los Alamos Nat Lab
Mainz Univ.
Manchester Univ.
Mol SCK/CEN
München TU
Nancy GRPG
Newcastle Univ.
Orsay JPN
Padova Univ.
Pavia Univ.
Rutherford RAL
Seattle (Univ. of Washington)
Sussex Univ.
Tübingen Univ.
Vienna Atominst.

## General Summary

The activities within College 3 cover a broad spectrum of interest. Studies range from fundamental physics experiments, the results of which can have important consequences for attempts to construct grand unified theories (GUTS), to the use of nuclear physics techniques as tools in such diverse fields as atomic physics and geology.

The thermal neutron induced fission process is studied using the parabolic spectrometer PN1 (LOHENGRIN) and the energy-time-of-flight spectrometer PN8 (COSI FAN TUTTE). Highlights in 1988 have included the discovery of a large odd-even effect in very asymmetric binary fission of $^{236}U$ (PN1) and the first ever study of the $^{229}Th(n_{th},f)$ reaction (PN8).

The importance of neutron capture reactions and the outstanding performance of the electron (PN2 - BILL) and gamma-ray (PN3 - GAMS, PN4) spectrometers continue to manifest themselves in an increasing demand for time on these instruments. One of the most important developments this year has been in the use of the GAMS4 spectrometer to measure excited nuclear state lifetimes (see blue box contribution). With the demise of OSTIS (SN6) last year nuclear structure studies of fission fragments are now performed only at PN1.

In the field of fundamental physics a particular effort has been put into precise measurements of the neutron beta decay lifetime. By early next year several independent groups will have completed such measurements using both bottle-type arrangements (based at the neutron turbine (TGV) on level D) and beam-type experiments (at the SN7 cold neutron beam position). Late in 1988 the neutron-antineutron oscillation experiment started to take its first data.

In addition to the instruments mentioned above several other neutron beams are used by the college. Topics studied include the interactions of low energy neutrons with various surfaces, neutron interferometry, parity violation in nuclear processes and gamma-gamma coincidences and correlations in nuclei following neutron capture. In addition the long standing activation analysis studies of geological and biological samples have continued.

## Scientific Trends and Highlights in 1988

### Fission

Since its discovery, 50 years ago, the fission process has been studied intensively via numerous reactions. Despite the effort going into this field of nuclear physics no established microscopic description of the fission process has emerged and today the characteristics of fission are still far from being predictable.

This was demonstrated in a convincing way during the last year on the fission product spectrometer LOHENGRIN (PN1) with the investigation of mass, nuclear charge and kinetic energy distributions for very asymmetric mass splits. Mass and energy distributions for fragment masses A = 69 to A = 86

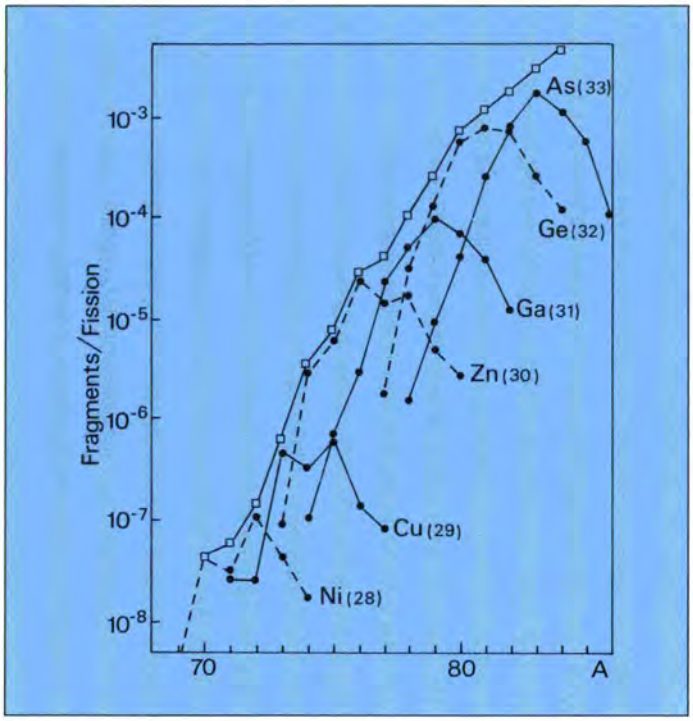



*Figure 1* *Isotopic (●) and mass (❑) yields for the very assymmetric fission of $^{235}U$. The large neutron even-odd effect is evident for the isotopic distribution of Ni, Cu and Zn.*

were measured for three targets: $^{235}U$, $^{239}Pu$ and $^{241}Am$ (collaborations with Orsay, Darmstadt and Mainz). The yields for these masses are about 5 to 9 orders of magnitude below the peak fission yield. For $^{235}U$, where the data evaluation is almost complete, a large and unexpected neutron pairing effect was discovered for the mass region A = 70 to A = 77. This odd-even neutron effect, which amounts to almost 80% for the Ni isotopes, has been shown to be dependent on the fragment kinetic energy (Fig. 1).

The search for both neutron induced cluster emission and the emission of heavy third particles from neutron induced ternary fission continued (Collaboration with Univ. of Tübingen). An initial investigation of the emission of $^{24,26}Ne$ and $^{28}Mg$ clusters from the $^{233}U(n_{th},f)$ reaction produced an upper limit for the decay rate of $\lambda \leq 1.8 \times 10^{-14} s^{-1}$ (for $^{24}Ne$). A successful search for ternary decay involving $^{24}Ne$ gave a branching ratio of ternary events to the total binary fission rate of about $10^{-12}$ for $^{233}U$.

The investigation of very neutron rich isotopes of Nickel and Copper recently observed, using PN1, for the first time (see Annual Report 1987) was continued. These nuclei are important because they are thought to lie on the path of the nuclear chart followed initially by the astrophysical r-process. In order to measure the half-lives of these newly observed isotopes a new detector system was included in the big ionisation chamber (BIC). A series of eight planar solid state detectors (of 500 μm thickness) were placed in the chamber, at the end of the ion trajectories. Using this set-up the time correlation between the incoming fission product and its subsequent β-decay electron was measured for the isotopes $^{72,74}Ni$ and $^{72,74}Zn$. As an example the decay curves for $^{72}Ni$ and $^{75}Cu$ are shown in Fig. 2 (collaboration with Orsay, Darmstadt and Grenoble University).

As part of the long standing effort on PN1 to accumulate systematics of fission fragment mass, nuclear charge (Z) and kinetic energy distributions for accessible actinide targets, the investigation of $^{241}Am(2n_{th},f)$ was completed (collaboration with Darmstadt). Furthermore, due to the Z-resolving ability of the big ionisation chamber, it was possible to complete a thorough measurement of the $^{229}Th(n_{th},f)$ reaction (collaboration with Los Alamos Nat. Lab.). For this latter nucleus the important proton odd-even effect indicated by earlier ΔE-E measurements (carried out at the neutron guide installation S10) was confirmed (Fig. 3).

On the fission fragment spectrometer COSI FAN TUTTE (PN8) correlated fragment mass, kinetic energy and nuclear charge distributions for the thermal neutron induced fission of $^{229}Th$ have been measured (collaboration with CEN Bordeaux). During the same run the $^{239}Pu(n_{th},f)$ reaction was studied for

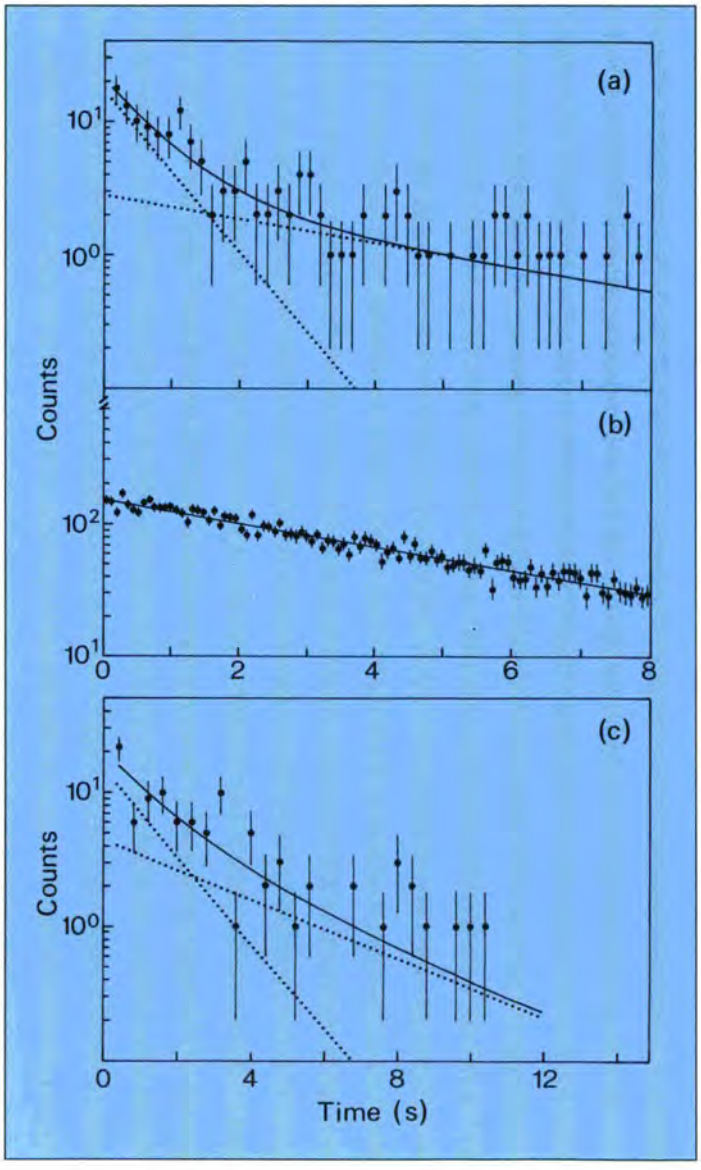



*Figure 2* *(a) and (c) show the spectra of time interval distributions between incoming ions and first detected β-ray. (b) shows a spectrum of random time interval distribution to be used in the analysis of correlated events.*

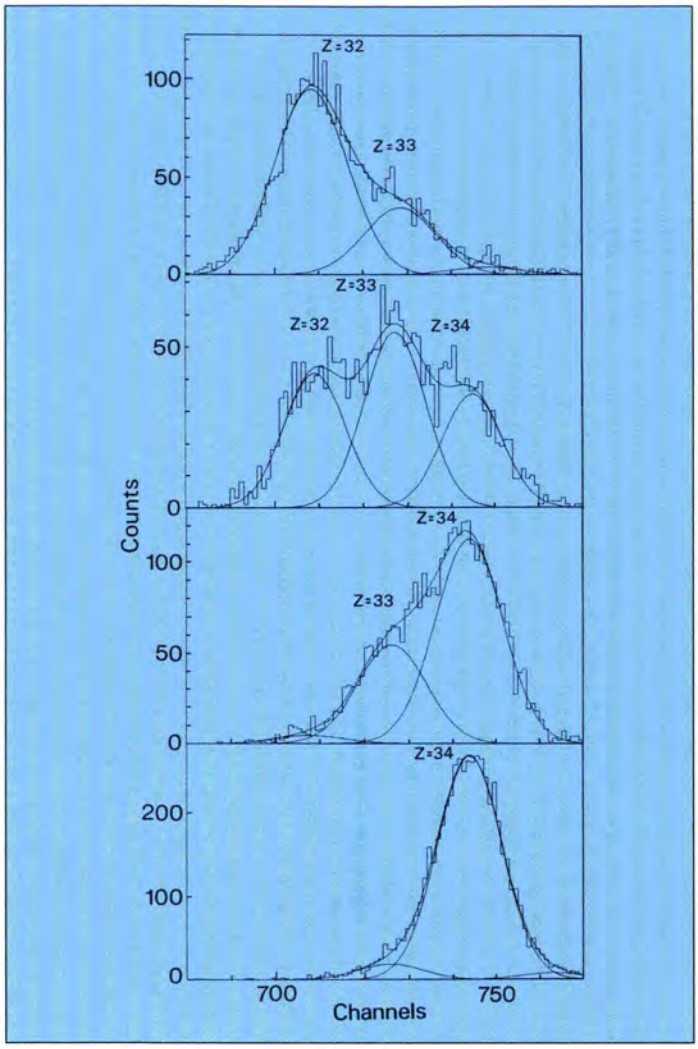



*Figure 3* *The nuclear charge distribution for four consecutive masses in $^{229}$Th. The enhancement of Z = 32 and Z = 34 over Z = 33 is very prominent.*

calibration and cross check purposes. The excellent resolution of the time-of-flight devices and the axial ionization chamber made it possible (for the first time) to make an unambiguous study of the characteristics in the light fragment region of the $^{229}$Th($n_{th}$,f) fissioning system, where individual masses are resolved by the spectrometer. Since the data analysis is still in progress only preliminary results can be given. Mass and kinetic energy distributions, for the light fragment group, were extracted rather easily from the extremely large list-mode data set. An average light-fragment mass $\langle M_l \rangle$ = 87.6 amu and a mean light-fragment kinetic energy $\langle E_l \rangle$ = 97.8 MeV has been found.

Using a derived Bragg-curve-spectroscopy method, developed at PN8, the nuclear charges of the light fission fragments were also determined for the two fissioning systems mentioned above. Their contributions to the mass yields are shown in Figs 4 and 5. A clear enhancement of the yields for the two even charges Z = 34 and Z = 36, from the $^{229}$Th($n_{th}$,f) system, has been found. From the integrated nuclear charge distributions quantitative odd-even effects of 39(4)% and 9(1)% can be calculated for the $^{229}$Th($n_{th}$,f) and $^{239}$Pu($n_{th}$,f) systems respectively.

One of the advantages of COSI FAN TUTTE is that it is non-selective in terms of any fragmentation of the fissioning system under investigation. This permitted a study (using the same data set as above) of so called "cold-fission" events, in the $^{230}$Th case. In Fig. 6 a mass distribution of "ordinary" fission (i.e. taking all kinetic energies into account) is shown, while Fig. 7 represents just "cold" events (i.e. having a light-fragment kinetic energy $E_l \geq 111$ MeV). The dominance of the cold fragmentation of the $^{230}$Th compound nucleus into masses 96 and 134 amu is due to the influence of strong neutron and proton spherical shells (N = 82, Z = 52) which combine to stabilize the heavy fragment at mass A = 134.

The $A_l/A_h$ = 86/144 mass split is, according to the shell-corrected scission point model, enhanced because the heavy fragment of mass A = 144 is favoured by a strongly deformed neutron shell (N = 88 and $\beta$ = 0.6).

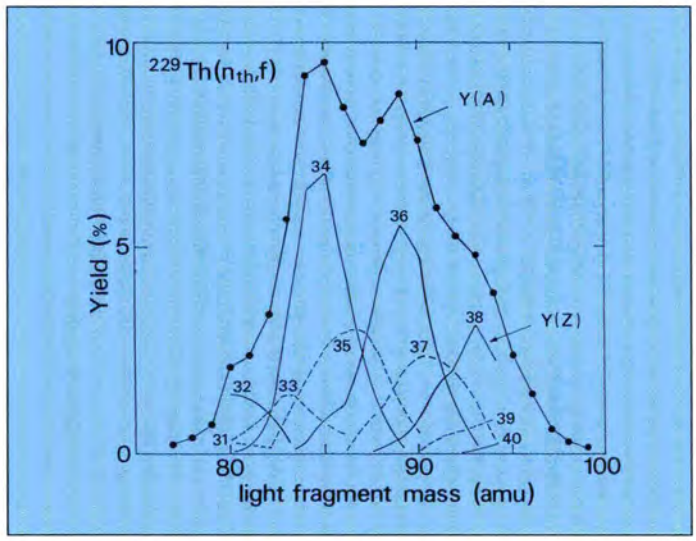



*Figure 4* *Distribution (integrated over kinetic energy) of nuclides as a function of light fragment mass for $^{229}$Th ($n_{th}$,f).*

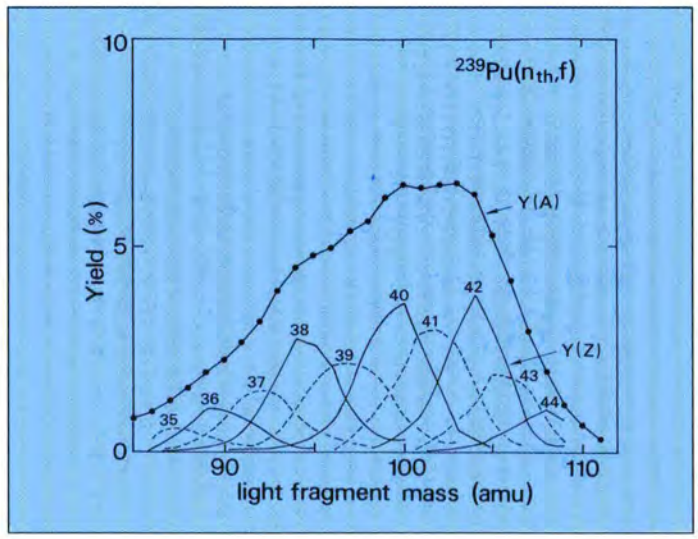



*Figure 5* *Distribution (integrated over kinetic energy) of nuclides as a function of light fragment mass for $^{239}$Pu ($n_{th}$,f).*

In order to verify the reliability of the complex data analysis in the $^{241}Pu(n_{th},f)$ studies at PN8 (collaboration with Univ. of Ghent and JRC Geel) extensive calibration measurements with a $^{235}U$ target have been performed. For this "classic" fission reaction the different fragment characteristics are well known and accepted. Good agreement was found between the PN8 results and those of PN1 as shown in Fig. 8, which represents the yield of each nuclide as a function of the light fragment mass. From this figure an odd-even effect (for $88 \leq A_l \leq 102$ amu and $33 \leq Z_l \leq 43$ ecu) of 28.2 (1.8)% was deduced for $^{235}U$, which agrees very well with the value of 27.7% derived from the PN1 data, and also with other values in the literature. However, a different behaviour of the proton odd-even effect as a function of the kinetic energy has been found for the $^{241}Pu(n_{th},f)$ system (Fig. 9). The tendency to a more or less constant odd-even effect for low kinetic energies observed for $^{242}Pu$ can also be seen in the most recent $^{234}U$ data.

In the last cycle of 1988, at the time of writing, a first attempt was being made to study the "exotic" $^{232}U(n_{th},f)$ reaction.

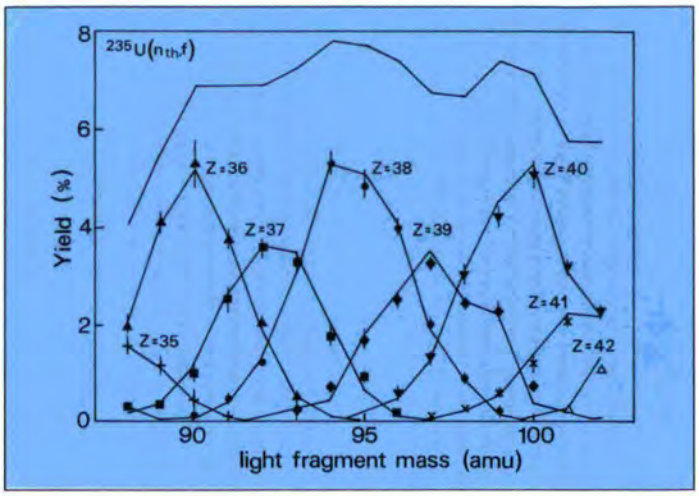



*Figure 8 Comparison between the distribution of nuclides as a function of light fragment mass in the $^{235}U(n_{th},f)$ reaction obtained at PN1 (full lines).*

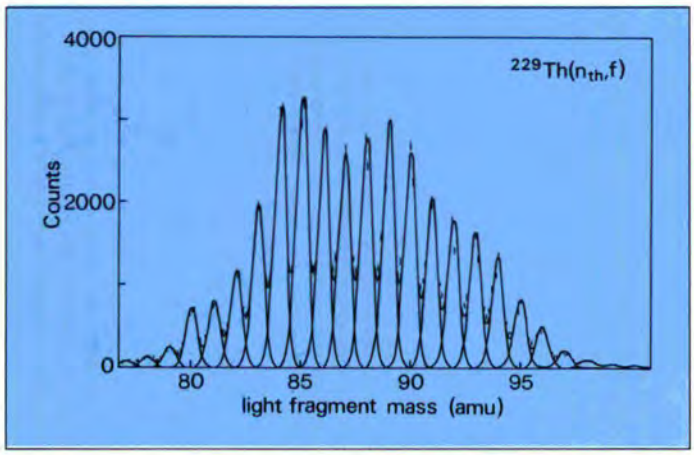



*Figure 6 Global light mass distribution for thermal neutron induced fission of $^{229}Th$.*

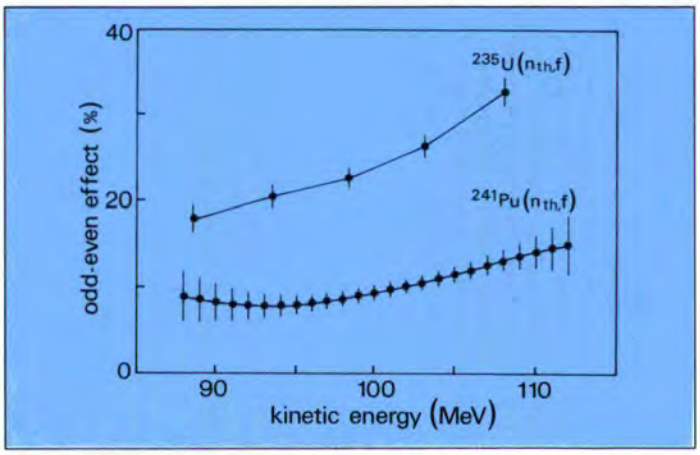



*Figure 9 Proton odd-even effect as a function of kinetic energy for $^{241}Pu$ $(n_{th},f)$ and $^{235}U$ $(n_{th},f)$.*

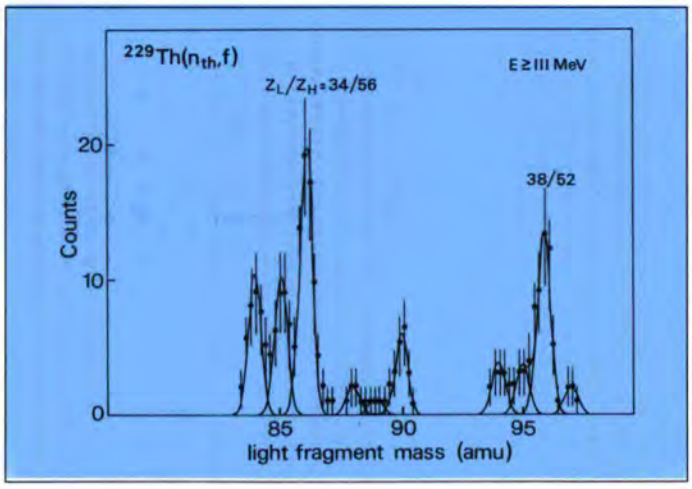



*Figure 7 Mass distribution for light fragments with $E \geq 111$ MeV in the $^{229}Th$ $(n_{th},f)$ reaction.*

## Nuclear structure

Nuclear spectroscopy studies at the ILL centre around the electron spectrometer PN2 (BILL), the crystal gamma ray spectrometers PN3 (GAMS 1, 2, 3 and 4) and the triple-coincidence pair spectrometer PN4. Though nuclear structure studies still provide the main source of experiments on these instruments their versatility is demonstrated by increasing activity in related fields.

In the field of nuclear structure the tests of Interacting Boson Model (IBM) predictions have again prompted many experiments. Some of these experiments have as their aim the construction of very detailed level schemes. Others are devoted to more specific nuclear structure problems which require the study of a few specific transitions.

This year very detailed conversion electron spectroscopy was carried out for the even-even nucleus $^{188}Os$ (collaboration with Univ. of Manchester and Univ. Tübingen). The new results, together with the $\gamma$-ray spectra previously obtained with the bent crystal spectrometers (GAMS 1, 2, 3), will lead to a much

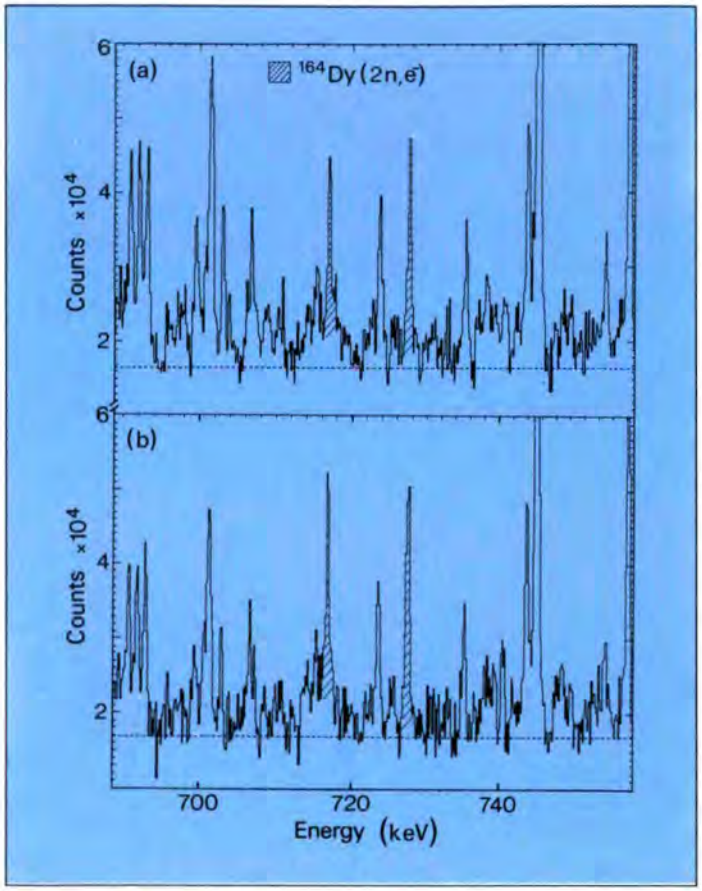



*Figure 10 A portion of the conversion electron spectrum following thermal neutron capture by $^{164}$Dy measured using PN2 a) shortly after insertion of the target into the neutron flux and b) after the double capture reaction has reached saturation. Peaks from the $^{164}$Dy (2n, e$^-$) reaction are indicated.*

better knowledge of this nucleus, which lies in the interesting transition region from the O(6) to the SU(3) limits of the IBM.

Electron and gamma spectra over a large energy range were also measured for the Dysprosium isotopes $^{165,166}$Dy (collaboration with Univ. of Tübingen). Since $^{165}$Dy is unstable, with a β-decay half-life of 2.4 hours, $^{166}$Dy must be studied following double neutron capture by $^{164}$Dy. The problem in such a measurement is one of assigning the observed transitions to either single or double capture and the studies mentioned above provide a good example of the two techniques available to achieve this. For the electron spectra this assignment relies on the different time dependence of the transition intensities, due to radioactive burnup. Therefore the spectra were measured twice - first just after the target was put into the neutron flux and then again with the $^{165}$Dy(n,e$^-$) reaction in saturation. Fig. 10 shows a part of these spectra with the lines from the $^{165}$Dy(n,e$^-$) reaction indicated. At PN3, however, the technique used to identify gamma transitions from single and/or multiple neutron capture is to perform measurements in different neutron fluxes. Fig. 11 demonstrates how easily these assignments can be made, by comparing spectra which have been recorded with a $^{164}$Dy target at different distances from the reactor core (55 and 75 cm respectively).

This recently developed facility can now be regarded as routine and another measurement of double neutron capture, on a $^{191}$Ir target, has recently been carried out (collaboration with Univ. of Fribourg). Analysis of the data from this experiment is underway.

In related work a very complete level scheme for $^{165}$Dy has been established (collaboration with SCK/CEN Mol) comprising all I = 1/2, 3/2 levels below 1.3 MeV. The level structure has been interpreted in terms of the Nilson model and quasiparticle-phonon coupling. In the excitation range from 500 to 1300 keV five gamma-vibrational bands and one octupole vibrational band have been identified by comparison of the (n,γ) data with particle transfer reactions.

Among the experiments looking at particular transitions the study of F-spin (isospin for a system of proton and neutron bosons) forbidden M1 transitions between collective levels was completed with measurements of the $^{161}$Dy(n,e$^-$) and $^{187}$Os(n,e$^-$) reactions (collaboration with Univ. of Köln). The results gave new information on E2/M1 mixing ratios for transitions in the gamma band and also yielded mixing ratios of 1 to I-1 gamma to ground band transitions.

A closely related topic is the study of the decay of the collective 1$^+$ states in deformed nuclei, which were first discovered by inelastic electron scattering. These were interpreted as a small amplitude oscillations of the angle between the neutron and proton symmetry axes (a so-called scissors mode). In the meantime these levels have been extensively studied with electron scattering and nuclear resonance fluorescence and much information about their location, their fragmentation, and the M1 strengths involved, has been collected.

However, almost nothing is known about their decays to other excited levels. On the basis of IBM-2 calculations it is expected that the decays to low lying collective 2$^+$ states will also occur via strong M1 transitions. Thus mixing ratios of these transitions are of great interest. Such data is very difficult to obtain since these 1$^+$ levels lie typically at ≈ 3 MeV excitation energy where the level density is high. A further problem is that the level energies are still not precisely enough known to identify their decay lines in very complex conversion electron or gamma-ray spectra. Nevertheless first attempts were made to study the situation in $^{158}$Gd (collaboration with Univ. of Sussex) and $^{164}$Dy (collaboration with Univ. of Tübingen) using PN2 and PN3 respectively. These continuing projects demonstrate nicely a situation often experienced in nuclear spectroscopy.

This is that only the detailed and very precise spectroscopy possible using the bent-crystal and conversion electron spectrometers allows the construction of complex level schemes, up to relatively high excitation energies, which are necessary to access the few crucial transitions of interest.

In a similar way sometimes crucial nuclear model tests can be made by looking at absolute transition probabilities from one or two particular levels of interest. In order to obtain these absolute values knowledge of the level lifetimes is essential. It has been shown recently that Gamma-Ray-Induced Doppler broadening (GRID) can be used to deduce short lifetimes (<$10^{-11}$ s) of excited nuclear states using the almost ideal resolution of the two-axis flat crystal spectrometer GAMS4. In this method lifetimes are compared to the slowing down time

of recoiling atoms in the target material (for details see blue box contribution). Systematic studies of the stopping behaviour have been carried out in some mono-atomic polycrystalline systems, such as metallic Ti, Cr and Fe, and diatomic systems, such as TiC and NaCl. Detailed tests of the respective atomic potentials needed to describe the atomic collisions are currently under way.

Another class of transitions of particular interest for discriminating between nuclear models are E0 transitions in even-even nuclei, which can be measured only with the electron spectrometer PN2. A recent publication about monopole transitions in $^{172}$Yb, measured with BILL, discusses a level scheme of this nucleus which includes five excited $0^+$ states and 13 E0 transitions. Such extensive data provides a very stringent test of theoretical predictions. In 1988 this program was continued with measurements of the $^{189}$Os(n,e$^-$) and $^{179}$Hf(n,e$^-$) reactions (collaboration with Univ. of Sussex).

The nuclear level schemes of odd-odd nuclei are among the most complex encountered experimentally because of the extra degrees of freedom provided by the presence of two unpaired nucleons. Yet it is only these nuclei that offer the opportunity of investigating the interaction between an unpaired proton and an unpaired neutron at low excitation energies. In deformed nuclei this interaction manifests itself in the energy splitting of a Gallagher-Moszkowski pair of rotational bands and in the odd-even energy offset (or Newby shift) found in K = 0 rotational bands.

Neutron capture gamma-ray spectroscopy is a particularly important technique in such studies due to the high levels of sensitivity and resolution obtained to date. Since milligram quantities of essentially non-fissile target materials are required, these detailed spectroscopic investigations, using the (n,γ) probe, are limited to just a few of the odd-odd actinide nuclei. Among these an important example is the recent study of $^{238}$Np which has provided significant improvements to the level scheme due to greatly improved sensitivity and resolution (collaboration with Lawrence Livermore Nat. Lab.). This has enabled the assignment of spins and parities to a large number of levels and allowed the construction of a rather complete level scheme below 600 keV, including four K = 0 bands.

A study of the $^{103}$Rh(n,γ) and $^{103}$Rh(n,e$^-$) reactions (collaboration with Imperial College London and Univ. of Göttingen) was also partly motivated by the rapidly emerging interest in odd-odd nuclei, this time in the context of new theoretical developments such as extended Boson-Fermion supersymmetries. This investigation should also provide new information about more general nuclear properties like level densities and especially gamma-ray strength functions, which have to be discussed in terms of statistical models. To this end both γ-rays and conversion electrons have been measured up to 1 MeV transition energy. This will lead to a vastly extended level scheme of that nucleus.

The spectrometers PN2 and PN4 have, this year, also been used to try and determine more precise energy calibrations for fundamental experiments being performed at other laboratories. At PN2 a high precision measurement of the 31 keV isomeric transition in $^{83}$Kr was performed (collaboration with Lawrence Livermore Nat. Lab.). This will be used to determine a precise energy calibration around the tritium beta-decay end point energy for use in a spectrometer at LLNL which measures the electron neutrino mass via the shape, close to the end point, of the beta decay spectrum from gaseous tritium. At the PN4 spectrometer an attempt was made to measure the neutron binding energy of $^{76}$Ge following double neutron capture by $^{74}$Ge (collaboration with CEN Bordeaux). The measurement of this value is needed in order to try and resolve possible ambiguities in double beta decay measurements of $^{76}$Ge to $^{76}$Se carried out by a Bordeaux-Saragosse-Strasbourg collaboration. This project will continue in 1989.

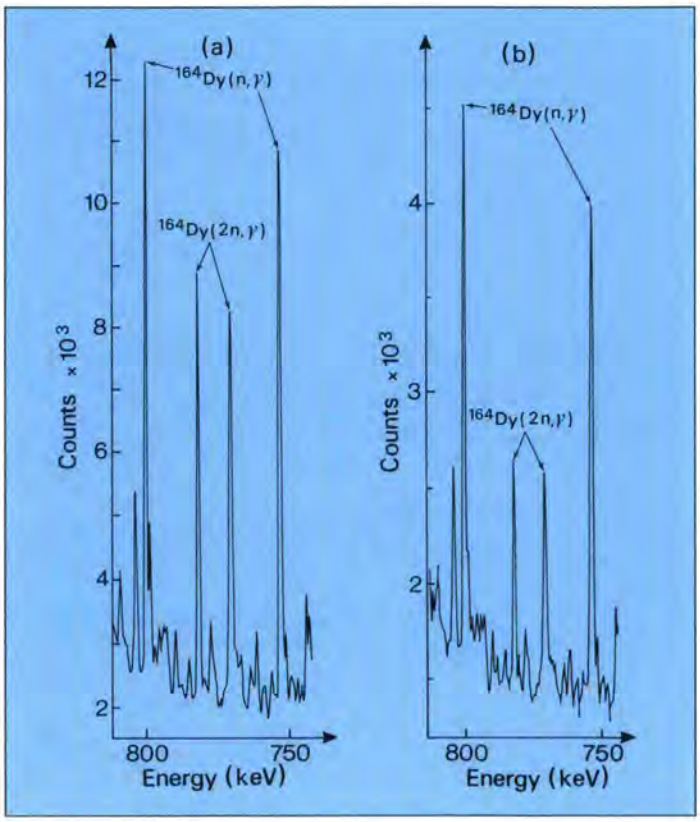



*Figure 11 A portion of the gamma-ray spectrum following thermal neutron capture by $^{164}$Dy recorded in first order of reflection using the GAMS 3 bent crystal spectrometer in neutron fluxes of a) 5.5 x 10$^{14}$ s$^{-1}$cm$^{-2}$. b) 3 x 10$^{14}$ s$^{-1}$cm$^{-2}$. The different flux dependence of the lines from single and double capture can be clearly seen.*

A major topic at BILL in 1988 was again the search for neutral resonances in Bhabba scattering (collaboration with GSI Darmstadt). Throughout 1986-88 the experimental set-up has been continuously improved (see technical section). The latest experiments have focussed on a search around the invariant mass of 1.8 MeV/c$^2$ which corresponds to one of the most prominent e$^+$e$^-$ pairs observed in heavy ion collisions at GSI. In a first run a positron flux of 8 x 10$^5$ e$^+$/s over a 10 x 100 mm$^2$ section of the focal plane was achieved. Scattering from a Beryllium foil was studied with an eight detector device in the energy interval from 2.1 to 2.4 MeV in 5 keV steps. For each energy about 4 x 10$^4$ Bhabba events were collected and within the statistical accuracy of 0.5% no resonance enhancement was detected. From this upper limits of 12.6 beV for the energy integrated total cross section and 3.8 meV for the resonance

width were determined. At the same time a group from Stuttgart reported a resonance from Bhabba scattering at 1.83 MeV/$c^2$ with a total cross section of 63 b.eV. The PN2 measurement was therefore repeated with improved statistics (positron flux now 1.2 x $10^6$ $e^+$/s). In total 1.6 x $10^5$ Bhabba events were collected and again no deviation from a smooth excitation function was observed. From this a new upper limit of 6.3 b.eV for the total cross section was derived, which definitely rules out the results from the Stuttgart experiment. This limit is the most stringent bound derived until now (in this mass region) from Bhabba scattering. It should be noted that the sensitivity is still only limited by statistical accuracy and thus could be improved by longer measuring times or a stronger positron source. However the limit already corresponds to a minimum lifetime of 3.5 x $10^{-13}$ s and thus experimental techniques which favour longer lifetimes are currently being tested.

## Fundamental physics

The research effort into fundamental physics at the ILL goes mainly into precise determinations of the properties of the neutron itself. More precise values determined for the neutron beta decay lifetime ($\tau_\beta(n)$) can have consequences for both astrophysics and weak interaction theory while possible observation of a neutron electric dipole moment (EDM) or of neutron-antineutron oscillations would provide tests and parameter constraints for grand unified theories (GUTS).

Whilst the latter of these projects has required the construction of a special installation other activities revolve around ultra cold and very cold neutron beams (UCN, VCN) provided by the neutron turbine (TGV) and cold neutrons from the SN7 guide, which can provide polarised, unpolarised and chopped beams.

The "bottle" neutron lifetime experiment (using UCN from the neutron turbine) mentioned in last years report was completed in 1988. Ultra-cold neutrons were confined in a glass box of variable length which was coated with a low reflection-loss fluorinated oil. Storage times of up to 730 s (corresponding to bottle lifetimes of greater than 4000 s) have been measured. The remaining wall reflection losses are eliminated by variation of the size of the storage volume, under simultaneous conservation of the number of wall collisions following a simple scaling law of the confinement times (Fig. 12). This relative method needs no support from additional experimental or computational input. Systematic errors are studied experimentally by varying the bottle length, surface structure, temperature, pumping time and pressure, as well as the incoming neutron spectrum. An experimental error of < 0.5% has been obtained from various runs with different experimental parameters. The final value of $\tau_\beta(n)$ will be published after completion of the data analysis.

A neutron life time measurement (at the SN7 neutron guide position) using a helium filled drift chamber in a monochromatic, pulsed, neutron beam (see Annual report 1987) provided a first result. The ratio of the decay electrons to the (n,p) reaction rate (from the admixed $^3$He) determined directly the neutron life time $\tau_\beta(n)$. The ionisation tracks of both types of events were recorded, including the ionising power. Initial information on the background below the decay electron events was obtained from the time structure of the event rate relative to the time-of-flight of the neutrons. A more realisitic determination was achieved by comparing the electron tracks in different parts of the drift chamber (Fig. 13). With three short runs a value of $\tau_\beta(n)$ = 886(30) s was obtained, where the uncertainty is dominated by the statistical accuracy. Major improvements are possible, both in background rejection and beam intensity (ILL-ISN-LAPP collaboration).

Using a different approach a research collaboration from the University of Sussex and NIST (U.S.A.) is, at the time of writing, running another experiment on the free neutron lifetime, again at the SN7 beam position. In this method the β-decay protons are trapped, for a fraction of a second, in a magnetic and electric field and then evacuated towards a proton detector. An almost background free measurement of the decay protons is achieved by this technique. The decay volume can be varied by changing the length of the trap and thus eliminating end effects. The neutron flux density is measured by a precisely calibrated $^{10}$B(n,α) detector arrangement.

In the long standing search for a neutron EDM data taking has continued in the last year using an essentially unchanged experimental set-up (i.e. a 5 litre storage volume and an electric field of 10 to 15 kV/cm). A preliminary analysis of the data gives an electric dipole moment of (-1.2 ±0.6) x $10^{-25}$ e.cm. Work is now underway on the development of a much larger storage volume, of around 50 l, and the use of a gaseous magnetometer, which would share the same storage volume as the neutrons. To this end a deuterated polystyrene surface has been investigated and found to be promising (see ILL technical report 88LA01T).

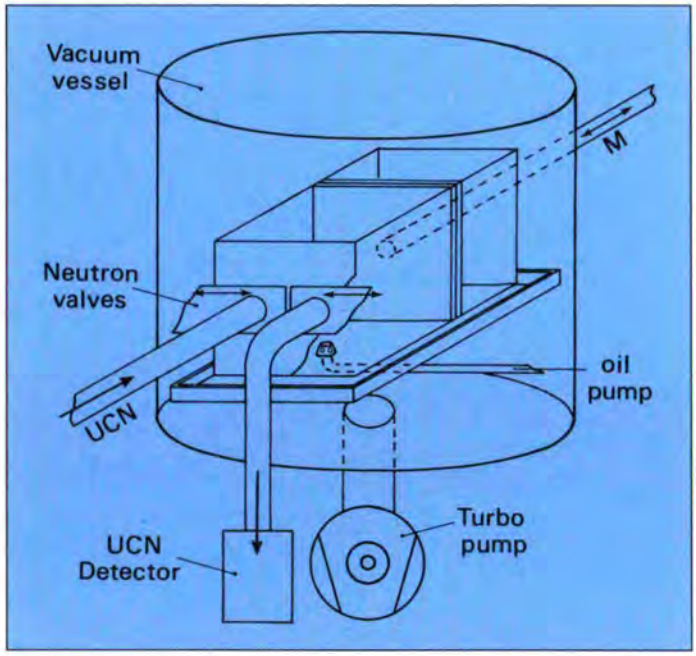



*Figure 12* *Schematic view of a neutron lifetime experiment using storable ultra-cold neutrons; the variable storage volume is a glass container with a movable rear wall. The UCNS enter and leave through sliding gate valves. The inner walls of the container are periodically coated with a fluorinated oil giving excellent UCN reflection characteristics and sealing properties to all mechanical parts.*

# College 3

During the first 2 reactor cycles of 1988 the neutrons extracted directly from the vertical cold source by the curved guide were used, after monochromatisation (1 neV < ΔE < 30 neV), for various experiments. This was done using the gravity monochromator (see photograph) constructed by D. Richardson (Univ. of Sussex thesis student) and allowed the measurement of the spectral distribution of the neutron sources themselves. Other measurements included the determination of the Fermi potentials of various materials, the energy dependence of UCN storage times and searches for quasi-elastic UCN scattering. This monochromator has made feasible a whole new set of experiments with UCN which were previously not possible.

Following this research the installation of the new, long baseline, interferometer for 100 Å neutrons (proposed by the Vienna-Munich group) was begun. First time of flight and interference measurements have been performed.

Using polarised neutrons, from the SN7 beam, a trial experiment was made to observe parity violation in neutron induced fission of $^{233}U$.

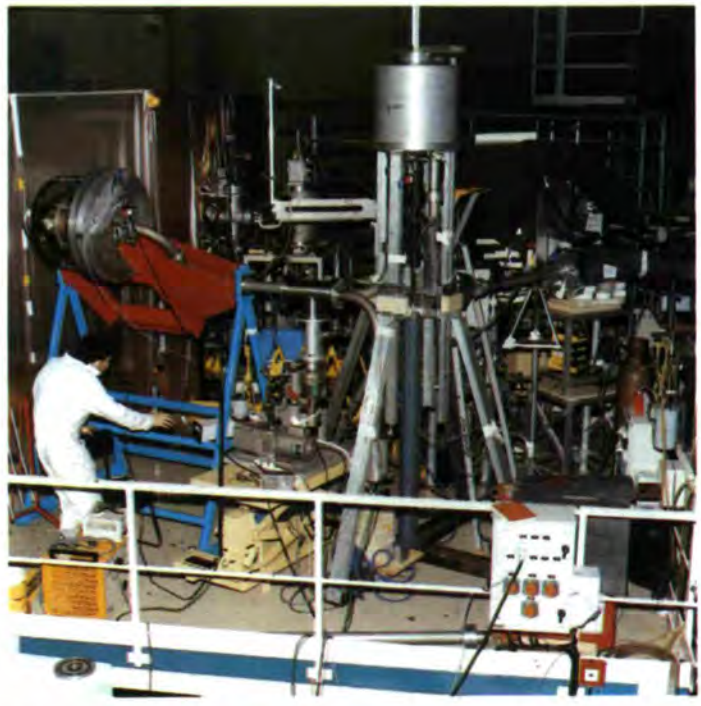
This would manifest itself in an asymmetry of the emission of fission products relative to the neutron spin direction. A high resolution twin ionisation chamber, where the common cathode carries the fissile target, was used to resolve the individual fission products. Following this feasibility test a full scale experiment is planned for 1989 (Collaboration with Univ. of Tübingen, ITEP and Kurchatov Moscow).

The neutron-antineutron oscillation experiment finally became operational during the last cycle of the year and started taking its first data. The photographs show a view of the annihilation product detector, with part of the cosmic ray veto shield opened and a close-up of the particle detector planes.

## Special beam instruments

College 3 has three positions at the end of the H22 thermal neutron guide (H22D/S10, H22E/S51 and H22F/S34). These are used for a variety of experiments which fall under the broad umbrella of the college. In the past year a number of interesting experiments have been carried out at these positions.

A research group from CBNM Geel (Belgium) has precisely measured the number of fission neutrons emitted in the $^{235}U(n,f)$ reaction as a function of the energy of the fission



*The gravity monochromator at the "level D" of the HFR.*

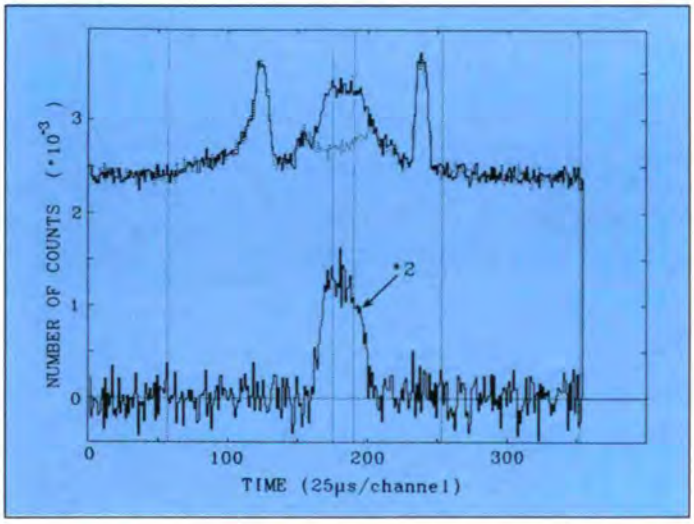



*Figure 13 Determination of the neutron life-time with a He filled drift chamber. The figure shows the electron tracks in different parts of the chamber.*

*Part of the flight path for the $n\bar{n}$ experiment. It leaves the second guide-hall (left of the picture) and enters into the detector hall (on the right).*

inducing slow neutrons. An energy dependence of this quantity, η, (as has been indicated by former measurements) would enter into the temperature coefficient of the reactivity of nuclear reactors. The present experiment was performed at both the SN7 beam (cold neutrons) and S34 beam (thermal neutrons) in order to cover a wide range of sub-thermal neutrons. Two slightly asynchronously running choppers and a neutron flux density monitor were used to define a monochromatic, pulsed, neutron beam by time-of-flight. By this TOF method the beam sweeps continuously through the neutron energies of interest. The beam struck a $^{235}U$ sample, which was thick enough to absorb all the fission inducing neutrons. The emitted neutrons from fission were counted by an NE213 liquid scintillator. In a separate measurement the shape of the neutron energy spectrum was determined by replacing the $^{235}U$ sample by $^{10}B$ or Cadmium and recording the emitted γ-rays. The data are under evaluation.

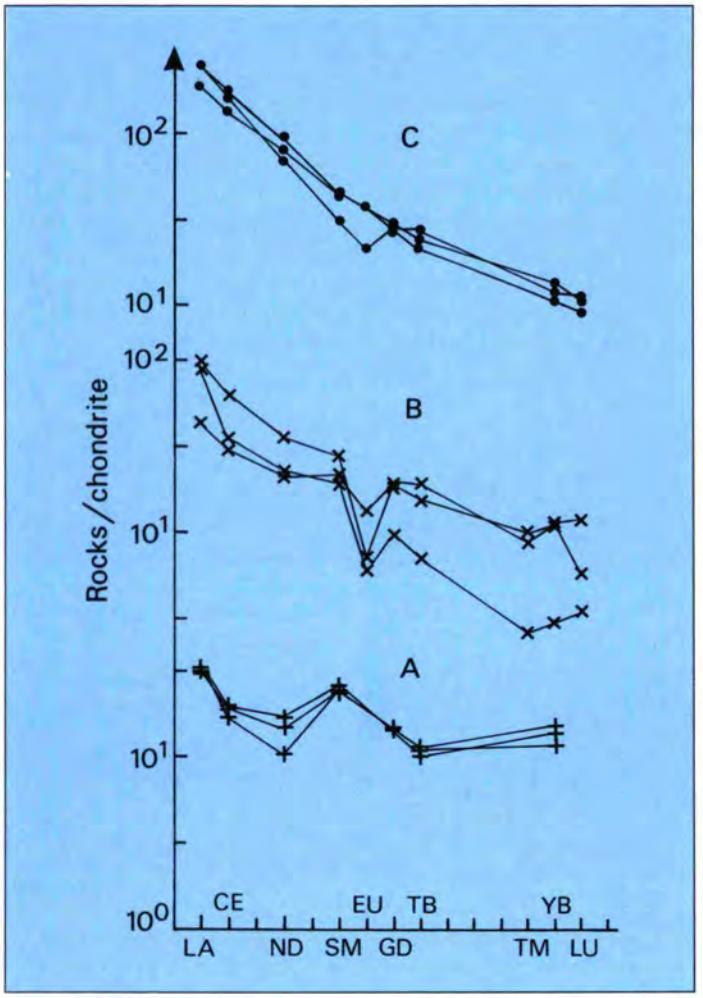



*Figure 14 Neutron activation analysis of various rock samples.*

Within the standard solar model the principal uncertainty in the production of high energy (Hep) neutrinos arises from a lack of knowledge of the rare $^{3}He(p,e^{+}\nu)^{4}He$ reaction rate, which is directly related to the $^{3}He(n,\gamma)^{4}He$ cross section.

However, previous measurements of this cross section are not in agreement (values range from 27 to 60 μb) and so a group from Argonne National Laboratory has performed an experiment at the S51 neutron guide position to try and obtain a firm value for this quantity. Using a large, well shielded, NaI detector and a mixed gaseous Helium and Nitrogen target the cross section can be deduced from a comparison of the 20 MeV capture γ-ray counting rate to the rate from the well known $^{14}N(n,\gamma)$ reaction. The data from this experiment are still being analysed but a preliminary result of around 55 μb is in agreement with two of the three previous measurements.

An ILL-University of Grenoble collaboration are regular users of the S51 position for prompt gamma activation analysis (PGAA) of geological samples. Of particular interest are

*The gigantic detector of the nn̄ experiment in the detector hall.*

measurements of the concentrations of Boron, Chlorine, Samarium and Gadolinium as part of a study into the role these elements play in the generation of leucocratic granitic magmas. This group also undertakes instrumental neutron activation analysis (INAA) studies of samples irradiated for a short time in the high neutron flux available near the reactor core. In this context studies have been made this year of Alpine/Variscan metabasic rocks from the Mont Blanc/Aiguilles Rouges massif (collaboration with Univ. of Fribourg), within plate basaltic rocks from the North Tibetan Plateau (collaboration with Univ. of Newcastle) and 1800 million year old greenstones from the central African Republic (Fig. 14).

The S34 special beam position is primarily used to perform gamma-gamma coincidence and directional correlation measurements on nuclei formed in the (n,γ) reaction. Regular users of this facility are a group from the University of Sussex, whose activities are currently focussed on a search for particular low-lying states, in even-even nuclei, which might have a 'mixed-symmetry' character when described in terms of the Interacting Boson Model (IBM). The experimental measurement of $^{200}$Hg mentioned in last year's report has led to a very complete study of this nucleus in terms of the IBM, including the probable identification of several such states. This project continued in 1988 with a study of the $^{179}$Hf(n,γ) reaction.

The nucleus $^{176}$Lu has been extensively studied at ILL, at both PN2 and PN3, due to its astrophysical importance (see Annual Reports 1986 and 1987). To complete this study a Belgrade-Karlsruhe-ILL collaboration has performed γγ-coincidence measurements at the S34 position in order to put parts of the level scheme on a more firm basis. A measurement was also performed to investigate subnanosecond lifetimes of excited states in this nucleus, using the centroid shift technique. To this end both γ, γ and γ, e$^-$-coincidences were recorded. The data are still being evaluated.

## Seminars etc...

Of particular note in the continuing seminar program are the twice yearly reviews, given before the Scientific Council subcommittee meeting, of various activities and instruments within the college. This year the two topics reviewed were:

i) fundamental physics activities using the neutron turbine and ii) the crystal gamma-ray spectrometers (GAMS).

Plans were finalised for a workshop on 'Fundamental Physics with Neutrons' to be held at ILL in the spring of 1989.

Finally three of the college graduate students (I. Förster, J. Copnell and D. Richardson) completed their theses in 1988. We wish them success in their future careers.

Secretary: Steve Robinson

# Determination of Short Lifetimes of Excited Nuclear States using γ-ray Induced Doppler Broadening

**H.G. Börner**

The determination of lifetimes of excited states in nuclei yields information about absolute transition rates from these levels. These are important quantities for sensitive tests of nuclear models and are therefore vital for the understanding of nuclear structure. There exist several, sometimes very complicated, methods which have been applied in the past encompassing a huge range of lifetimes from the attosecond region to many seconds. Fig. 15 shows a schematic overview of the direct methods used to date.

During the last two years a new method has been developed at ILL in which ultra-high resolution γ-ray spectroscopy is used for extracting lifetimes of levels populated after thermal neutron capture and subsequent γ-decay: the target is placed in the centre of the throughgoing beam-tube H6-H7 in a neutron flux of 5.5 x $10^{14}$ $cm^{-2}s^{-1}$. Thermal neutrons are captured by the target nuclei, the capture states of which decay via primary γ-transitions of energy $E_o$. These are isotropically distributed and induce recoils (initial velocities $V_R$ of the recoiling nuclei), which are in turn, isotropically distributed. The recoiling atoms are slowed down in the target material due to atomic collisions with the surrounding atoms. The energies ($E_1$) of the γ-ray from the subsequently populated states emitted during the slowing down period are Doppler-shifted. These transitions are measured with high resolution using the two-axis flat crystal spectrometer GAMS4 [1] (installed in a collaboration NBS-ILL). As one measures the projection of the velocity vectors onto the axis of observation defined by the spectrometer (effective solid angle ~ $10^{11}$) one measures a Doppler broadening with a maximum broadening

$$2\Delta E_{max} = \frac{2E_0 E_1}{Mc^2}$$

for $V = V_R = E_o/Mc$.

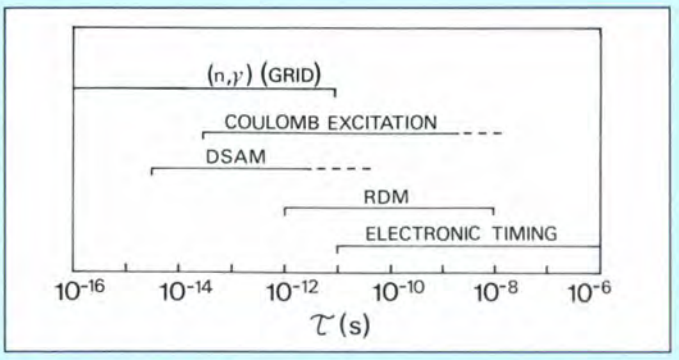



*Figure 15 Ranges of lifetimes of excited states covered by GRID compared to some other commonly used methods.*

The recoil velocities induced by primary γ-rays are ~ $10^{-4}$ to $10^{-5}$ c and consequently the maximum Doppler broadening $2\Delta E_{max}$ is very small.

As an example: for A = 49, $E_o$ = 5 MeV, $E_1$ = 1.5 MeV we obtain $\Delta E_{max}$ = 163 eV. This has to be compared with the very high resolving power of GAMS4 which is today typically about 30 eV (20 ppm) for 1.5 MeV γ-rays. The final measured line profile depends on the velocity distribution of the recoiling atoms in the sample. This is governed by the initial recoil and subsequent collisions with the neighbouring atoms and, evidently, by the lifetimes of the levels involved. To extract the lifetimes of the excited states one needs a description of the kinetic energy loss $dE_{kin}/dr$ during the slowing down process of the recoiling atoms in the target. A systematic study of the stopping behaviour in different materials is under investigation. Currently a model is used (and tested experimentally) where the atom loses its kinetic energy $E_{kin}$ by elastic collisions with the target atoms [2,3].

The measured line shapes originate from a convolution of the instrumental response function and the Doppler profile. An important characteristic of the two crystal geometry is that there exists a non-dispersive and a dispersive mode of measurement. In the non-dispersive mode (Fig. 16, $+\theta_B$-mode, left side) both crystals measure in the same reflection order such that the reflection planes are parallel to each other. The profile obtained by rocking the second crystal is the RESPONSE FUNCTION of the instrument. When the second crystal is placed at $-\theta_B$ (Fig. 16, right side) the instrument operates in a dispersive mode and as such allows the measurement of the ENERGY-DISTRIBUTION of the observed γ-ray. Thus the Doppler-broadening due to the recoil can be directly determined.

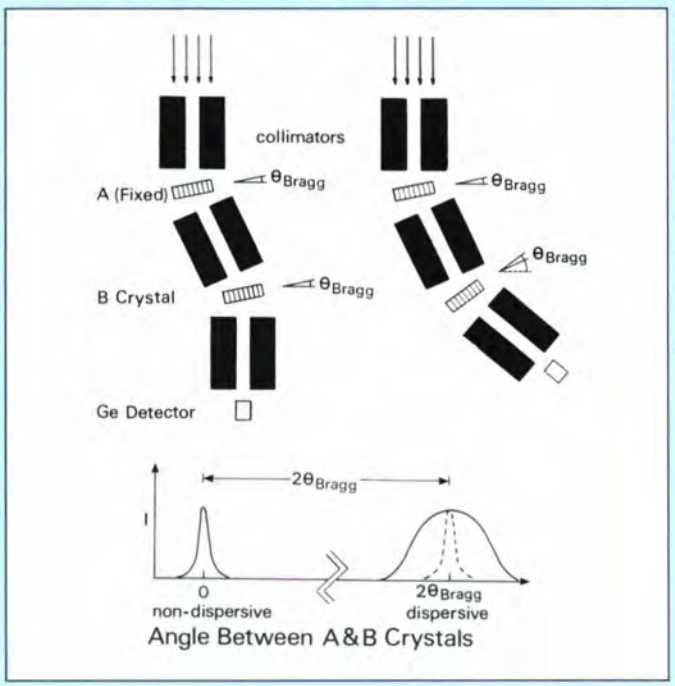



*Figure 16 Schematic diagram illustrating the two axis flat crystal method.*
*Left: non dispersive mode;*
*Right: dispersive mode.*

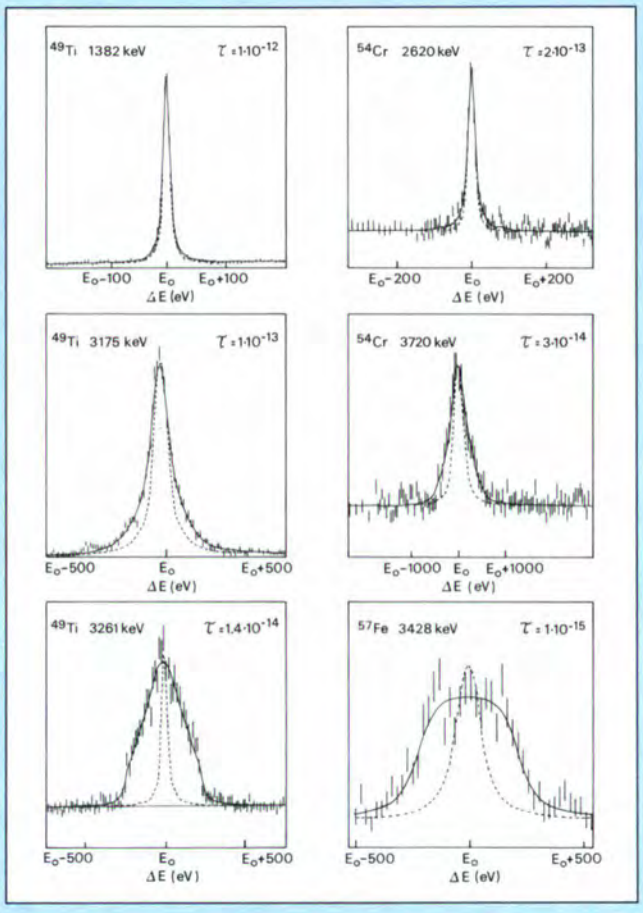



*Figure 17 Line shapes obtained for transitions depopulating levels with lifetimes ranging from pico seconds to femto seconds. The examples shown for $^{54}Cr$ [4] led to the determinations of lifetimes which were crucial in interpreting certain levels in this nucleus to be of mixed symmetry in the context of the Interacting Boson model IBM 2.*

Fig. 17 shows an ensemble of different transitions with lifetimes ranging from the $10^{-12}$ to $10^{-15}$ s regions, clearly indicating the change of the profile as a function of the lifetime of the excited states. These transitions have been obtained from nuclei in the region around A = 50.

## References


[1] *E. G. Kessler, G. L. Greene, M. S. Dewey, R. D. Deslattes, H. G. Börner, F. Hoyler. Inst. Phys. Conf. Ser. N°88, p. 167.*

[2] *H. G. Börner, J. Jolie, F. Hoyler, S. Robinson, M. S. Dewey, G. Greene, E. Kessler, R. D. Deslattes, Phys. Lett. B, 215 (1988) p. 45.*

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# Structural and Magnetic Excitations

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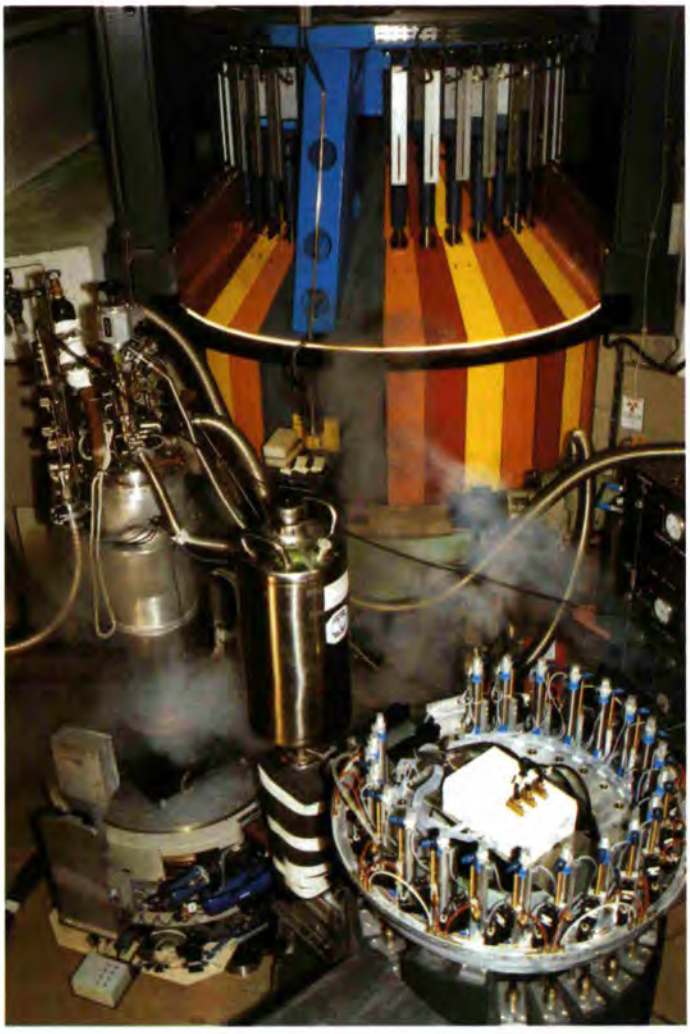
| M. Vallade | (U.S.M.G.) |
| P. Monceau | (CRTBT, CNRS) |

## General Summary

Research activity in college IV during 1988 took place in a wide range of fields centred around magnetic and structural excitations in solids. The college considered proposals for the three-axis spectrometers (IN1, IN3, IN8, IN14 and IN20), the time-of-flight machines (IN4, IN5, IN6, D7), the spin-echo machine (IN11) and the small-angle instruments (D11, D17). The beginning of 1989 saw the withdrawal of IN3 and the start of scheduled experiments on the new three-axis spectrometer IN14.



*Birth of the new triple-axis spectrometer IN14 at the horizontal cold source.*

## Scientific Trends and Highlights in 1988

### Lattice Dynamics and Structural Phase Transitions

#### Phonons in bcc metals

The investigation of phonons in the high temperature bcc phase of group 4 metals continued [1]. From the technical point of view these measurements are difficult to perform because these metals undergo a martensitic phase transition at temperatures above 860°C, thus single-crystals have to be grown and orientated on the spectrometer.

The complete phonon dispersions of bcc Ti and bcc Zr and their temperature dependence over the whole existence range of the bcc phase have now been measured (Fig. 18). The phonon dispersion for both metals behave in a similar way and the frequencies scale with the homology rule.

The dispersion curves are dominated by two striking features, both of which mirror the tendency of the group 4 metals to undergo martensitic phase transitions.

1) The transverse [**110**] branch with [**110**] polarization (known as the $T_1$ branch) is of very low frequency. This is directly related to the bcc-hcp martensitic phase transition which is achieved by the displacement corresponding to this branch at **q** = (0.5, 0.5, 0), the zone boundary. Fig. 19 shows how this so-called N-point phonon shifts to lower frequency as the phase transition is approached. The decreasing frequency is proportional to $1/u_o$ ($u_o$ = max. vibrational displacement).

2) The longitudinal branch propagating in the [**111**] direction is soft near (2/3, 2/3, 2/3) where scattered intensity is observed down to zero energy transfer (Fig. 20).

This so-called ω-phonon achieves the displacements which are needed to form the ω-structure. The ω-structure is a trigonal phase which is stable only under a pressure of several tens of kilobars pressure. Surprisingly, the frequency distribution at the ω-point does not change at all with temperature. Rather, we should interpret the frequency dip at the ω-point as a general weakness of the group 4 bcc structure which exists over the whole temperature range of this phase.

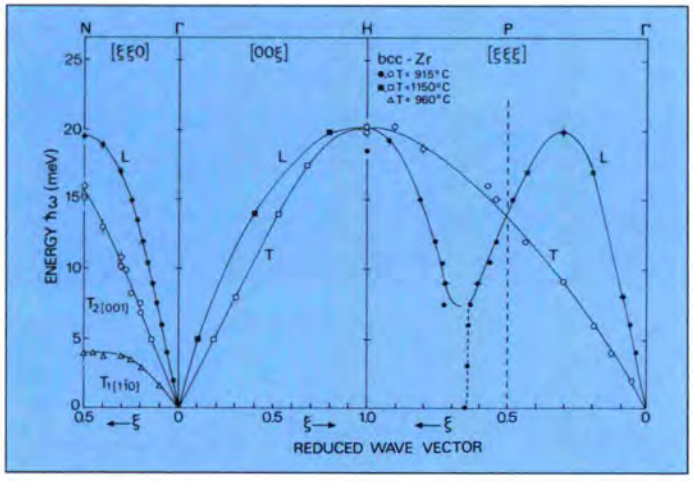



*Figure 18 Phonon dispersion of bcc Zr measured at approx. 915°C.*

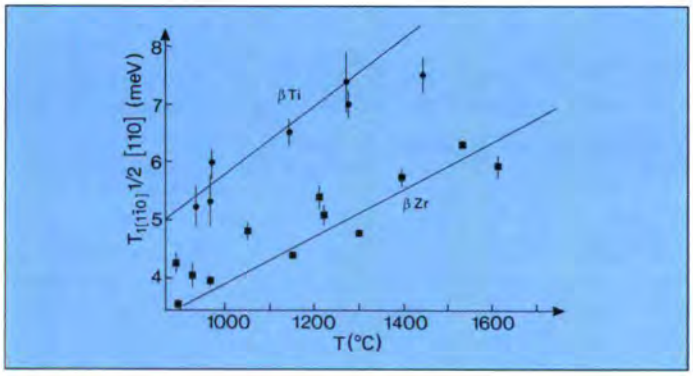



*Figure 19 Temperature dependent shift of the $T_1$ branch at q = 1/2[110] for bcc Ti and bcc Zr. The straight lines indicate the result of a common fit to both data sets related by the homology rule.*

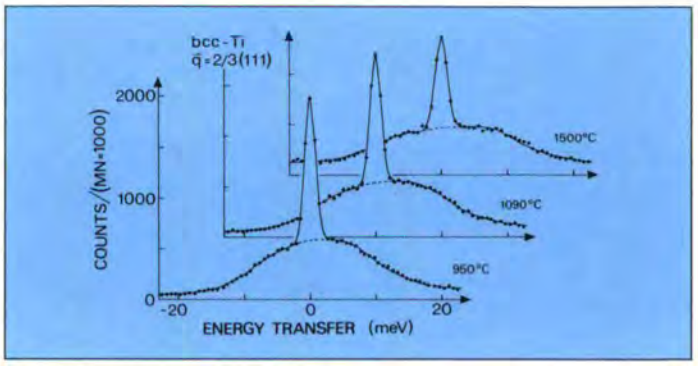



*Figure 20 Energy scans at q = (2/3, 2/3, 2/3) for bcc Ti at different temperatures. For neutron energy loss and gain broad shoulders of inelastic intensity are observed. Note that the elastic peak at hω = 0 can be entirely explained by elastic incoherent scattering events. The inelastic intensity has been fitted with a damped harmonic oscillator yielding identical results for all temperatures.*

# Magnetism

## High temperature superconductivity

This year has seen further work on the new "High Tc Superconductors". Their structural and magnetic properties are rich in physics. The lamellar copper oxides materials exhibit complicated and subtle phase diagrams when the hole concentration is varied. Magnetic interactions can provide large energy couplings, compatible with high Tc superconductivity. Although it is not clear yet whether the Cu spins play a role in the mechanism leading to the superconductivity, $La_{2-x}Ba_xCuO_4$, $YBa_2Cu_3O_{6+x}$ and the other cuprates materials have received considerable attention because magnetism and superconductivity seem to be intricately bound together.

The first studies of magnetic excitations in single crystals of $La_2CuO_4$ were made at Brookhaven [2]. At ILL these measurements have recently been extended up to higher energies using the hot source triple-axis spectrometer IN1 [3]. A magnetic response is observable up to energies of 0.1eV. Measurements at these energies afford the unique possibility of resolving, for the first time, the two spin wave branches. This may be seen in Fig. 21. $La_2CuO_4$ is a highly two-dimensional system so the spectrometer has been arranged so as to have its direction of poorest resolution along the $\mathbf{b}^*$ direction (the 2–dimensional axis). The data have been fitted to a model scattering function folded with the full resolution function of the spectrometer to yield values for the spin wave velocity of 800 ±100 meVÅ. Preliminary measurements on $La_{2-x}Ba_xCuO_4$, show that this value drops in doped samples.

In the $YBa_2Cu_3O_{6+x}$ system, the oxygen content can be easily changed from x = 0 to x = 0.93. Work has taken place at the ILL [4] on single crystals grown at the CEN-Grenoble using a grain-growth technique.

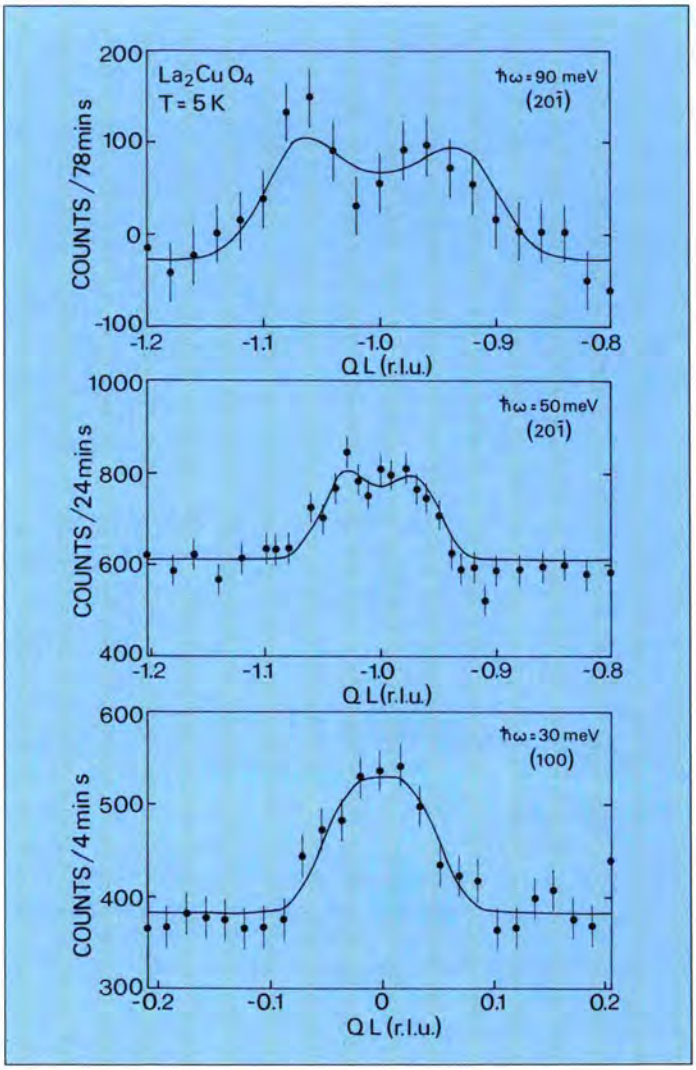



*Figure 21 Antiferromagnetic spin waves in $La_2CuO_4$, as measured on IN1[2]. The continuous lines are the result of a folding of the spectrometer resolution and a model scattering function for spin waves, this yields a spin wave velocity of 800 ±100 meVÅ. A measured background has been subtracted from the 90 meV data.*

Neutron diffraction experiments have revealed the existence of antiferromagnetic order of the $CuO_2$ planes with very high $T_N$ values, the moments on the Cu(2) sites being confined in the planes. In $YBa_2Cu_3O_6$ $T_N$ = 415 K and no ordered moment appears on the Cu chain sites. The two $CuO_2$ sheets of the unit cell are coupled antiferromagnetically and form magnetic bilayers. When the oxygen content is increased, the antiferromagnetic coupling of the bilayers is gradually destroyed. The disorder between bilayers is manifested by the appearance of rods of diffuse scattering perpendicular to $CuO_2$ planes. Close to $x_c$ = 0.40, some static disorder appears in the bilayers themselves. Above $x_c$, where superconductivity occurs, no long-range magnetic order can be observed (Fig. 22).

The large value of $T_N$ in these 2-dimensional systems is due to the large intra-plane exchange constants (J~1000 K). The exchange constant can of course be determined from inelastic neutron scattering. Fig. 23 shows constant Q scans made on relatively small-sized samples (0.4 $cm^3$ of $YBa_2Cu_3O_{6.2}$) [5] at various points along the line (0.5, 0.5, *l*). Two branches of excitations corresponding to the in-plane and out-of-plane fluctuations can be seen. The dispersion in the **c*** direction is very small, showing the nearly 2-dimensional nature of the magnetic interactions. The variation of the dynamic structure factor leads to a modulation of the intensity. By contrast, the in-plane dispersion is large (Fig. 23): the spin-waves are so stiff that the two branches originating from the magnetic zone centre cannot be resolved, even at large energy transfers (Fig. 23). From this data, the spin wave velocity can be estimated to be somewhere in the range 0.6-1.0 eVÅ. With increasing oxygen concentration x the peak in the constant energy scans becomes broader showing the spin wave velocity decreases with increasing oxygen content.

## Magnetic structure and excitations of amorphous ferromagnets

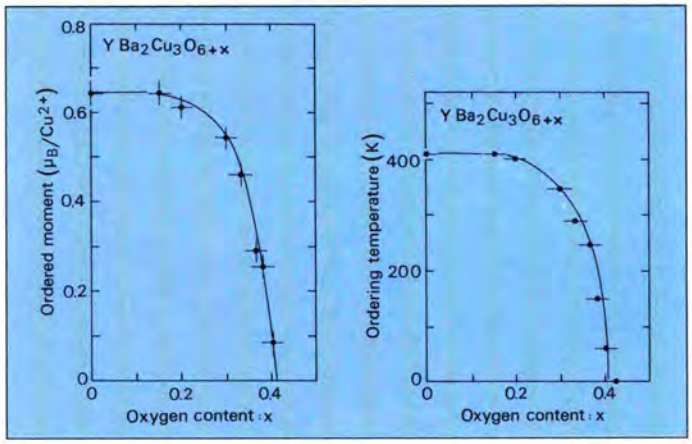



*Figure 22 Ordered moment and ordering temperature as a function of the oxygen content in $YBa_2Cu_3O_{6+x}$.*

The magnetic structure of amorphous ferromagnets is usually assumed to be one in which the magnetic moments are all aligned parallel to the spontaneous magnetization. This structure was tested during recent polarized neutron scattering experiments [6] with the triple-axis spectrometer, IN20, using polarization analysis for both incident and scattered beams. It was found that there was an appreciable spin-flip component to the elastic scattering, thus showing that the spins are not completely aligned. The wavevector dependence of the scattering (Fig. 24) is described by the magnetic form factor of the ions suggesting that the deviations in the spin directions are nearly randomly distributed from site to site.

The magnitude of the spin deviations varies from sample to sample. It is too small to be observed in a commercial METGLAS sample of $Fe_{78}B_{12}Si_{10}$ but large enough to be seen in a sample of $Fe_{83}B_{17}$, (Fig. 24). Measurements on samples containing Ni, $(Fe_{50}Ni_{50})_{78}B_{12}Si_{10}$ and $(Fe_{25}Ni_{75})_{78}B_{12}Si_{10}$, showed that the misalignment of the spins was substantially larger in these materials.

Fully polarized inelastic scattering measurements (Fig. 24) showed quasi-elastic spin-flip scattering which has relatively

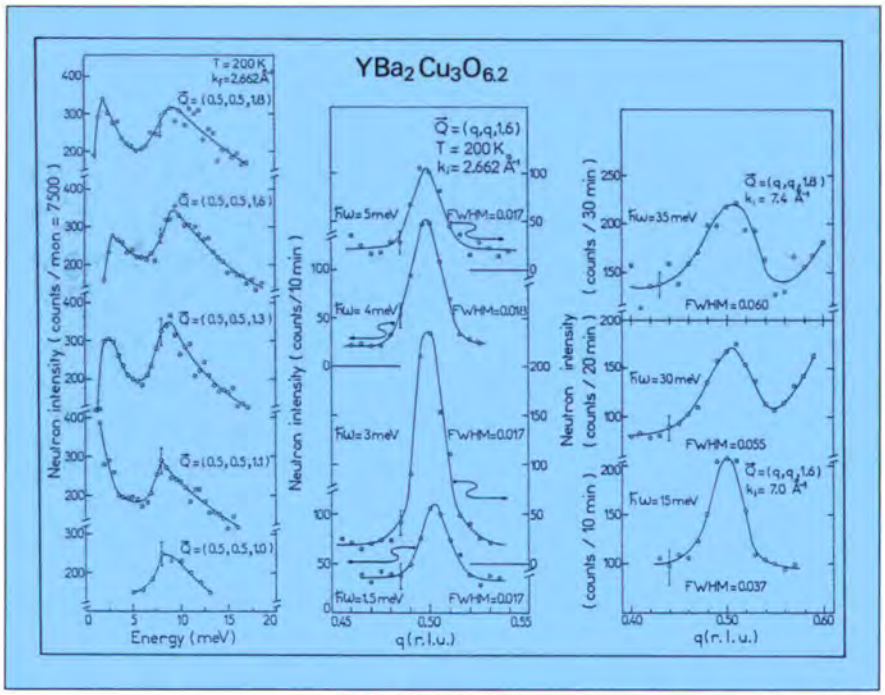



*Figure 23* *a) Constant-Q scans in $YBa_2Cu_3O_{6.2}$ at different positions along the c* axis.*
*b) In-plane Q-scans at different energy transfers in $YBa_2Cu_3O_{6.2}$.*
*c) High energy transfer scans in $YBa_2Cu_3O_{6.2}$.*

little wavevector dependence. These almost localized excitations may explain why the magnetization varies more rapidly with temperature in such amorphous systems than predicted from the measured spin wave stiffness constant.

## Dyon precession in $CsCoBr_3$

In 1986, it was predicted by Affleck [7] that solitons in antiferromagnetic chains should be the analogue of dyons, which in grand-unified field theories describe particles with electric (and/or magnetic) charge. In this model, while the mass of the particle is equivalent to the inverse width of the domain wall - the soliton - the charge would correspond to precessions of the spins inside the soliton.

The application of an external magnetic field H should therefore affect the frequency spectrum of the soliton modes. In the specific case of quantum spin chains close to the Ising limit, the effect of an external field has been explicitly calculated [8]. In zero field [9], the spectrum of the fluctuations induced by the soliton motion extends from frequency $\omega = 0$ to $\omega_M = 4\ \varepsilon J \sin q$, where a maximum is expected (J is the exchange coupling and $\varepsilon$ characterizes the departure from the pure Ising case ($\varepsilon J = J_x, J_y$)).

With an applied field (perpendicular to the spin direction in our case), a doubling of the soliton modes is predicted and two maxima should occur in the frequency spectrum. A study of this doubling effect has been performed using the compound $CsCoBr_3$ on IN8 [10]. It is well exemplified in Fig. 25 where the data obtained at the wavevector q = 0.5 r.l.u. for H = 0 and H = 9.8 T are shown. The lines are smoothed curves through the data, the background measured at low temperature (T = 1.4 K) having been subtracted. The arrows show the positions of the maxima calculated for H = 0 and H = 9.8 T. The agreement is very good. This inelastic neutron study complements a recent observation of the uniform (q = 0) soliton modes in a field by Electron Spin Resonance [11] and confirms the theoretical prediction of Affleck.

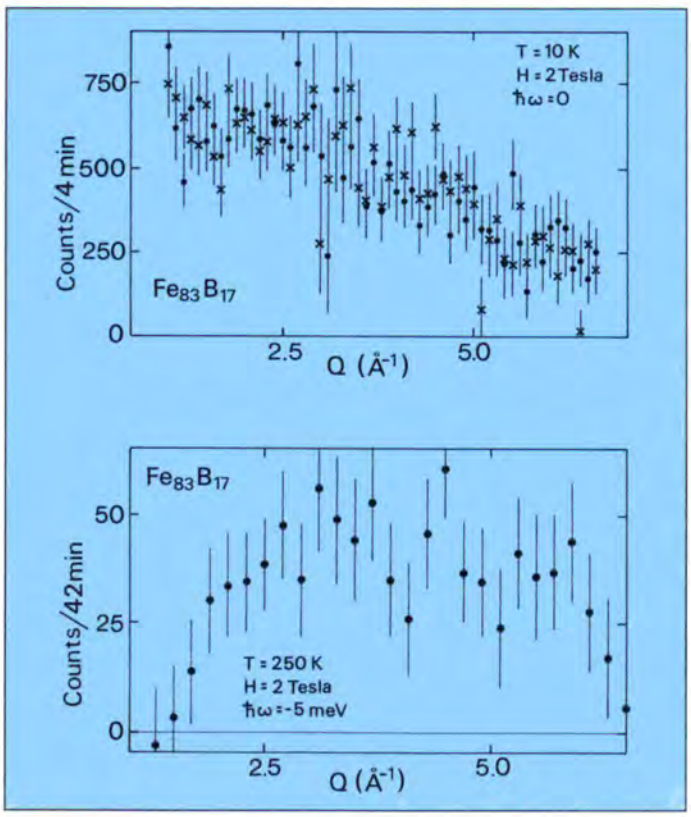



*Figure 24*
*(Top)* *Wavevector dependence of the magnetic scattering from $Fe_{83}B_{17}$ for H = 2 T and T = 10K. The data have been corrected for depolarization in the sample.*
*(Symbols : ● = spinup→ spindown, × = spindown → spinup)*
*(Bottom)* *Wavevector dependence of the inelastic spin flip scattering in $Fe_{83}B_{17}$ for $\hbar\omega$ = -5 meV (i.e. neutron energy gain). The figure shows the difference between the scattering at 250 K and a background at 10 K, with an applied field H = 2 T.*

## Magnetic excitations in $CsNiF_3$

In the one-dimensional magnetic system $CsNiF_3$, the spins show ferromagnetic correlations along the chain axis in the z-direction. Anisotropy makes this a planar system in which the spins are confined to the x-y plane. Above the three-dimensional ordering temperature $T_N$ = 2.8 K the fluctuations have the character of 1-D spinwaves along the chains. Excitation frequencies are observed due to in plane (IP)

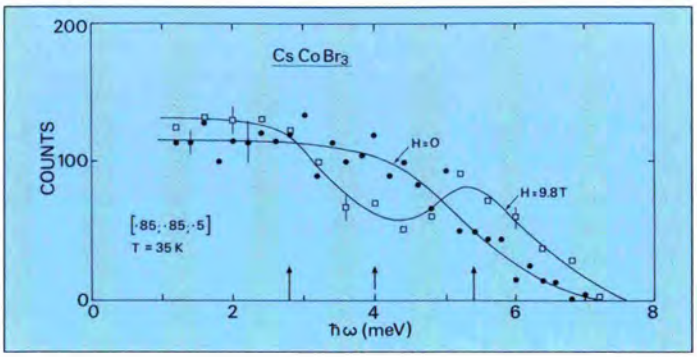



*Figure 25 The inelastic spectrum of $CsCoBr_3$ at q = (0.85, 0.85, 0.5) at T = 35K for H = 0 and H = 9.8T. The lines are smooth curves through the data. A background measured at T = 1.4 K has been subtracted.*

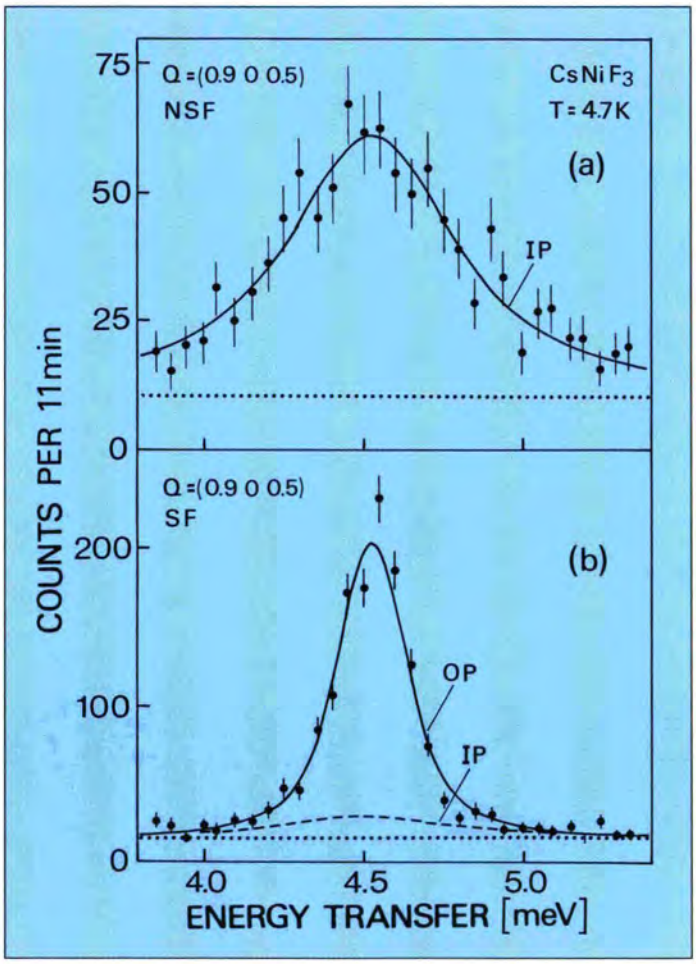



*Figure 26 Spin waves in $CsNiF_3$. Constant-Q scans are shown for the NSF and SF channels. The data have been taken at a position where $\alpha = 61°$ (see text). They are fitted with a Lorentzian line shape folded with a Gaussian of width 0.19 meV. The IP fluctuations give contributions to both channels. In the SF channel the IP signal contributes 24% of its intensity in the NSF channel.*

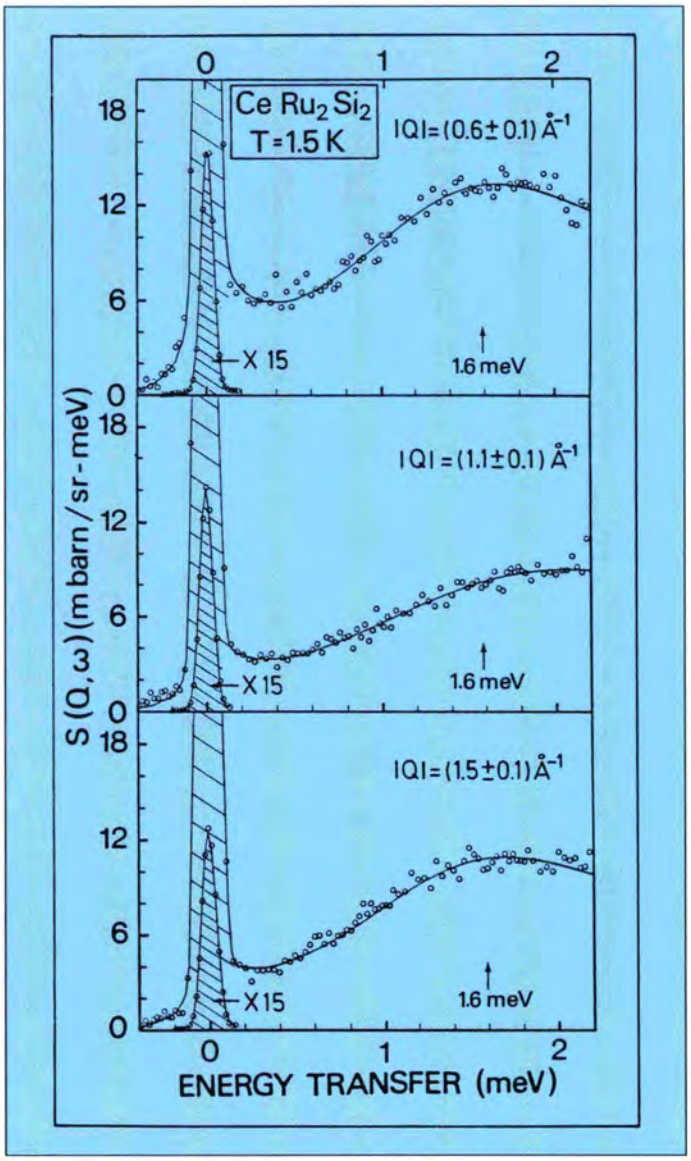



*Figure 27 Magnetic response of $CeRu_2Si_2$ at 1.5 K for three different |Q| values. The data have been fitted with inelastic Lorentzians.*

fluctuations, which have large amplitudes, as well as due to out of plane (OP) fluctuations with small amplitudes. The IP signal has a width corresponding to the correlation length along the chains, while the OP signal is much narrower.

A polarized neutron experiment with polarization analysis can be used to separate the OP signal and thus test theoretical predictions about its line width. If the sample is mounted such that the z-axis lies in the scattering plane and a small vertical field is applied, then the intensity due to the OP fluctuations is proportional to $\sin^2\alpha$ ($\alpha$ being the angle between **Q** and z), this signal only occurs in the spin flip (SF) channel. The IP fluctuations give contributions in both the spin flip (SF) and non-spin flip (NSF) channels, with the ratio of the intensities (SF/NSF) being proportion to $\cos^2\alpha$. Hence the signals due to the two different components can be separated as shown in Fig. 26 [12].

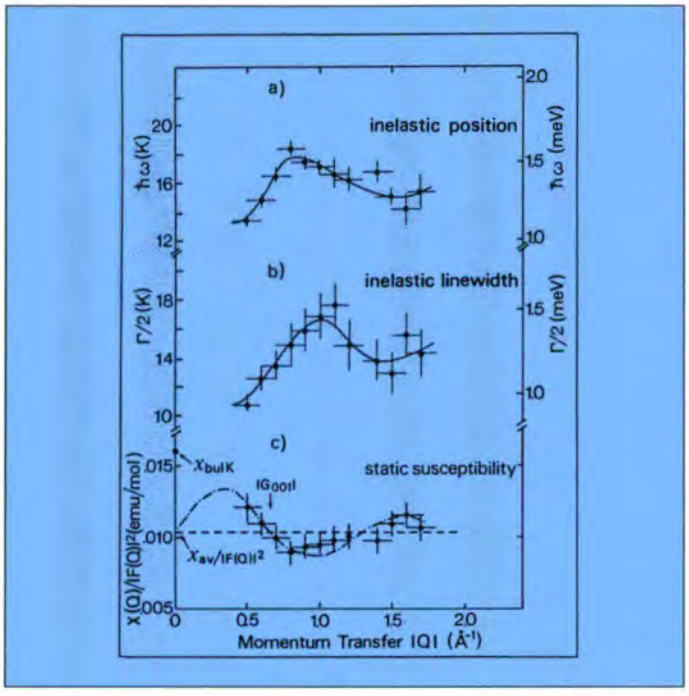



*Figure 28a Parameters obtained from fitting the data at 1.5 K: (a) inelastic position, (b) inelastic line width and (c) static susceptibility divided by the $Ce^{3+}$ magnetic form factor as function of |Q|. $\chi_{bulk}$ indicates the bulk susceptibility.*

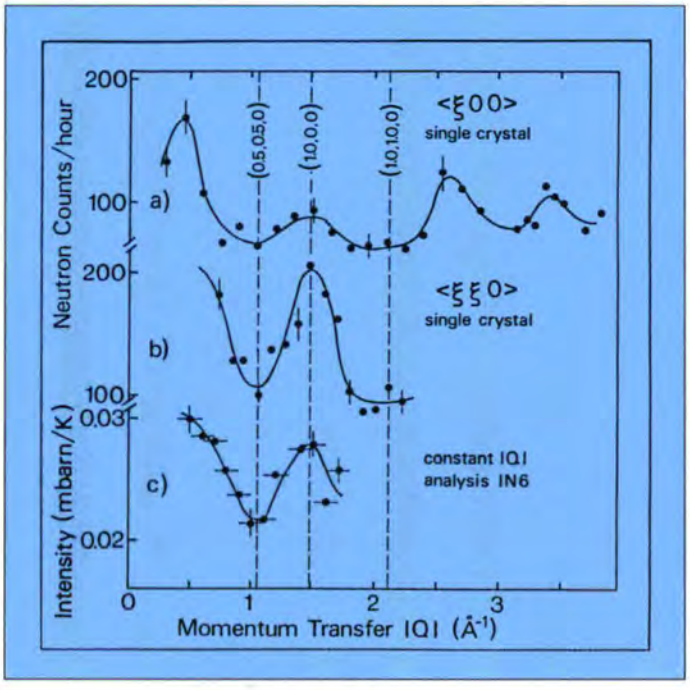



*Figure 28b A comparison of TOF and triple axis data: (a) and (b) are the constant energy scans of the single crystal experiment by Regnault et al. [5], (c) intensity of the inelastic Lorentzian at ħω = 1.6 meV resulting from the constant |Q| analysis of the powder data. Comparing (b) and (c) we can see the peak corresponding to Q = (0.7, 0.7, 0) in the TOF data.*

The data shown in Fig. 26 were obtained using the spectrometer IN20 [12] with $k_F$ = 2.662 Å$^{-1}$. These conditions, together with the slope of the spin wave dispersion produce very good instrumental focussing. If the excitations were sharp they would have a measured width of 0.19 meV. The intrinsic (Lorentzian) width of the signal due to the OP fluctuations was determined, for the first time, to be about 0.14 meV. It has a maximum of 0.18 meV at $q_c$ = 0.7 $q_{max}$.

## Magnetic excitations in heavy fermions

Neutron scattering techniques have proved to be a unique probe of the spin fluctuations in anomalous rare earth and actinide compounds. In particular heavy fermion materials have been the subject of much investigation. In these systems, the variation of the degree of hybridization of the 4f and conduction electrons leads to a wide variety of magnetic behaviour. We describe below the investigation of one compound $CeRu_2Si_2$ by two complementary inelastic neutron scattering techniques; triple-axis and time-of-flight.

$CeRu_2Si_2$ possesses the tetragonal $ThCr_2Si_2$ structure. The static susceptibility is strongly anisotropic ($\chi_{\parallel}/\chi_{\perp} \approx 15$) [13], the susceptibility for the c-axis follows a Curie-Weiss law at high temperatures, passing through a maximum at about 10 K and reaching a constant value as T tends to zero. The linear coefficient of the specific heat is strongly enhanced, $\gamma$ = 385 mJmol$^{-1}$K$^{-2}$ [14]. However, for temperatures down to 40 mK, neither superconductivity (as in $CeCu_2Si_2$) nor magnetic order have been observed [15].

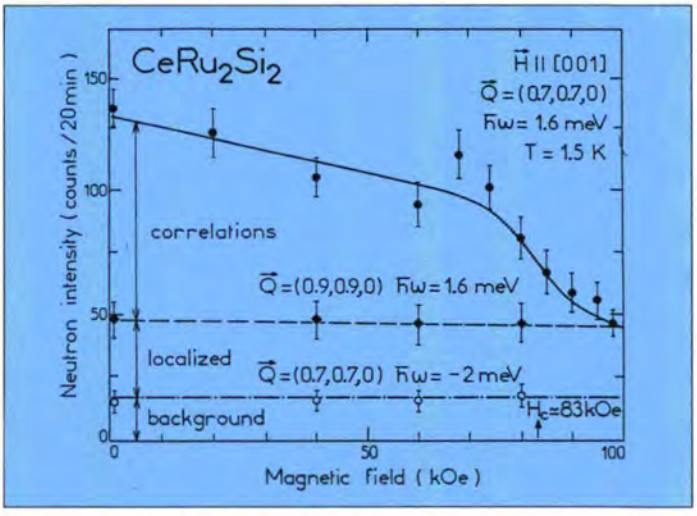



*Figure 29 Field dependence of the magnetic intensities measured at T = 1.4 K in $CeRu_2Si_2$. The correlations correspond to the peak maximum at Q = (0.7, 0.7, 0). The localized part has been measured at Q = (0.9, 0.9, 0).*

Measurements on a powder sample of $CeRu_2Si_2$ made using the time focussing time-of-flight (TOF) spectrometer IN6 [16] are shown in Fig. 27. The spectra shown are corrected for background scattering, absorption, detector efficiency and are calibrated with respect to a vanadium standard. The fits to the data take into account the strong variation of the resolution function with energy transfer and the magnetic form factor

F(Q) of $Ce^{3+}$. At 250 K the magnetic signal of $CeRu_2Si_2$ is quasi-elastic and can be fitted with a quasi-elastic Lorentzian with width, $\Gamma/2$ = 0 meV. With decreasing temperature the signal narrows and below 50 K, a |Q| dependence of the magnetic signal must be included to fit the data. Here a constant |Q| analysis has been performed. For temperatures below 20 K, the magnetic response changes from being quasi-elastic to inelastic. In Fig. 28 spectra for three different |Q| values at T = 1.5 K are shown. The spectra have been fitted with an inelastic Lorentzian where the position $\hbar\omega$ and line width $\Gamma/2$ are |Q| dependent. The resulting static susceptibility divided by the $Ce^{3+}$ form factor varies with |Q|, but the product of line width and static susceptibility is approximately |Q| independent.

The TOF data show the development of magnetic correlations with decreasing temperature. From triple-axis measurements [17], we find that the response is composed of two contributions: one **Q** independent (apart from the form factor) and the other peaked at positions with **q** = (0.3, 0, 0) and **q** = (0.3, 0.3, 0). The correspondence between the TOF data and triple-axis data is shown in Fig. 28. Fig. 29 shows the variation of the response at **Q** = (0.7, 0.7, 0) (this corresponds to **q** = (0.3, 0.3, 0), with applied magnetic field as measured on the triple axis instruments IN8 and IN20 [18]. At H = 8.3 Tesla the system undergoes a metamagnetic like transition. It can be seen from the neutron data that this is accompanied by a collapse in the magnetic correlations. At high magnetic fields the remaining response is then **Q** independent.

College Secretary: S.M. Hayden

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# "Magnetic" Systems Which Lose Their Magnetic Moment at Low Temperatures.

B. Dorner

The observation that some compounds are magnetic at high temperature and non-magnetic at low temperature is not new. This can be understood within the level scheme of a magnetic electron in the **crystal electric field splitting**. It may be that the groundstate is non-magnetic. The corresponding excitations can be observed with inelastic neutron scattering.

In this article we wish to present quasi one-dimensional substances with spin S = 1 in which the **exchange interaction** between neighbouring "magnetic" ions has a strong influence. Some of them have no magnetic moment at low temperature. The lowest crystal field splitting will be described by a **single ion anisotropy**. For the propagating excitations we will discuss in particular **the eigenvectors** (precession of neighbouring correlated magnetic moments).

The essential features of such materials are described by the Hamiltonian

$$H = -2J\sum S_i S_{i+1} + A\sum (S_z)^2 \qquad (1)$$

where J is the exchange parameter along chain direction and A is a single ion anisotropy parameter connected to the z-component of the spin direction. In this way the anisotropy determines a quantisation axis to be z. In all cases which are discussed in the following, it coincides with the structural z-axis.

A one-dimensional (1D) system as described by the Hamiltonian eq. (1) does not exhibit long-range order at finite temperature. Only correlations build up. But in reality quasi 1D materials show a phase transition to 3D order due to the interaction between neighbouring chains. The corresponding term in the Hamiltonian is left out for simplicity because it does not concern the discussion which will follow.

As a side remark we mention the Haldane conjecture [1], which concerns materials with integer spin values. This conjecture predicts an energy gap in the dispersion curve of the magnetic excitations due to spin fluctuations even if the anisotropy is zero.

We will consider the influence of A, in particular of its sign, on the magnetic structure and the magnetic excitations at **low temperatures**.

### A = 0

First we wish to recall the case A = 0, which is a **Heisenberg system.** A positive J leads to ferromagnetic correlations and a negative J to antiferromagnetic ones. The axes of their spin direction have no preferred orientation in the lattice. Magnetic excitations can be observed for finite wavevectors $q_z$.

For $q_z \rightarrow 0$ the frequencies extrapolate to zero. The dispersion relations are

$$\hbar\omega(q_z) = 4JS[1-\cos(cq_z)] \qquad \text{for } J>0 \qquad (2)$$

$$\hbar\omega(q_z) = 4JS\sin(cq_z) \qquad \text{for } J<0 \qquad (3)$$

c is the distance between neighbouring spins. Two neighbouring spins at low T for J > 0 can be taken parallel. Their motion for small $q_z$ is an in-phase precession around their spin orientation.

For J < 0 neighbouring spins can be taken antiparallel.

For small $q_z$ their precession is such that they maintain the antiparallel orientation. Fig. 30 shows that in a projection parallel to the spins the transverse amplitude rotates in **the same sense.** This means that relative to the spin direction one-spin precesses in a **right handed** and one in a **left handed** way. In a Heisenberg system with negative J there are two degenerate modes due to time reversal symmetry. As seen in Fig. 30 the two modes $\mathbf{e}_1$ and $\mathbf{e}_2$ have opposite sense of precession. These degenerate modes split up under an applied external magnetic field due to Zemann splitting.

### A < 0

Let us now consider a negative anisotropy parameter A. This defines the z-axis as an easy axis. The energy of the system is lowered if the spins are directed parallel or antiparallel to z. This means that quantum mechanical states m = +1 and m = -1 have equal energy (neglecting the exchange energy) and their energy is lower than for the state m = 0. Such a system is called an Ising system where the freedom of spin orientation is reduced to ±z. Any transverse amplitude in x or y direction costs energy. Therefore the spin wave dispersion curves exhibit an energy gap at q = 0. The eigenvectors are still as described in Fig. 30 (interchanging y and z). The antiferromagnetic excitations continue to be degenerate in the absence of an external field. There are not many substances known with negative A and S = 1. One example is $CsNiCl_3$ [2].

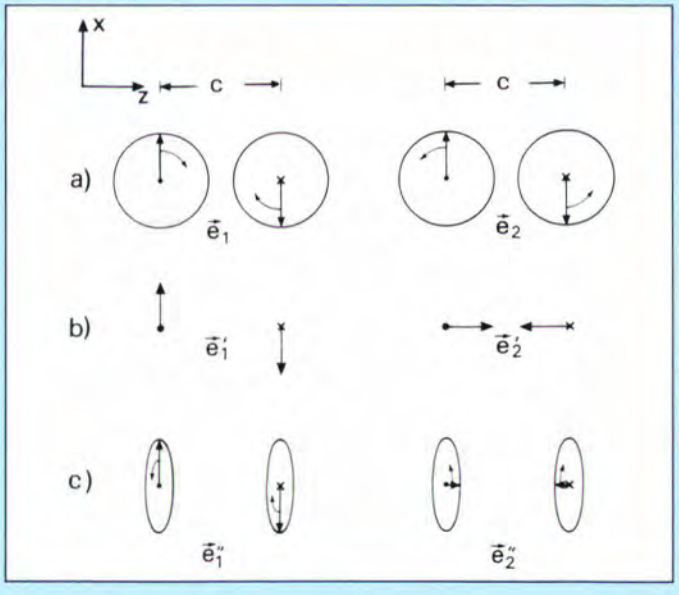


*Figure 30 Eigenvectors for a magnetic system with exchange parameter J < 0 in projection along the spin direction taken as y-direction. a) and b) Two equivalent sets of basic vectors for the degenerate spin waves in a Heisenberg System (A = 0). For an Ising System (A < 0) they are valid, if one interchanges the y- and the z-axes. c) gives the model eigenvector for $CsFeBr_3$: $\mathbf{e}_1''$ belongs to the low frequency mode and $\mathbf{e}_2''$ to the high frequency mode. Note that $\mathbf{e}_1''$ has antiferromagnetic configuration for IP and ferromagnetic ones for OP. In contrast $\mathbf{e}_2''$ has ferromagnetic components for IP and antiferromagnetic ones for OP.*

**A > 0**

A positive anisotropy makes the z-axis a hard direction. Now the m = ±1 states have a higher energy than m = 0. Neglecting for a moment the exchange energy, the parameter A gives the energy difference between m = 0 and m = ±1. In lowering the temperature to kT << A, **only states m = 0** will be occupied. This has the consequence that an ion with **spin = 1** in the state m = 0 **has no magnetic moment**. Disregarding exchange as done in the introduction, the susceptibility of such a system will go to zero at low enough temperatures and no magnetic correlations will appear. Excitations in such a "Singlet Ground State" (SGS) system must be called excitons. The system is in the **SGS-phase**, which is characterized by the quantization axis z.

If the exchange coupling is strong enough [3]

$$8|J| > A \qquad (4)$$

then it leads to **the new phase of the x-y system** characterized by a spin orientation axis in the x-y plane. The spins are free to orient in any direction perpendicular to z. Therefore the system is as well called a **planar Heisenberg system**.

If **J is positive** in such a x-y system, the dispersion of the spin wavesis given by [4]

$$\hbar\omega = 2S[(2J - 2J\cos(cq_z))\,(2J - 2J\cos(cq_z) + A)]^{1/2} \quad (5)$$

For $q_z \rightarrow 0$ the frequency still goes to zero because there is no preferred orientation in the x-y plane. This can be visualized if one looks to the eigenvectors. For any $q_z$ the spin does not any more precess on a circular one but on an elliptical one with the out-of-plane (OP) amplitude smaller than the in-plane (IP) one.

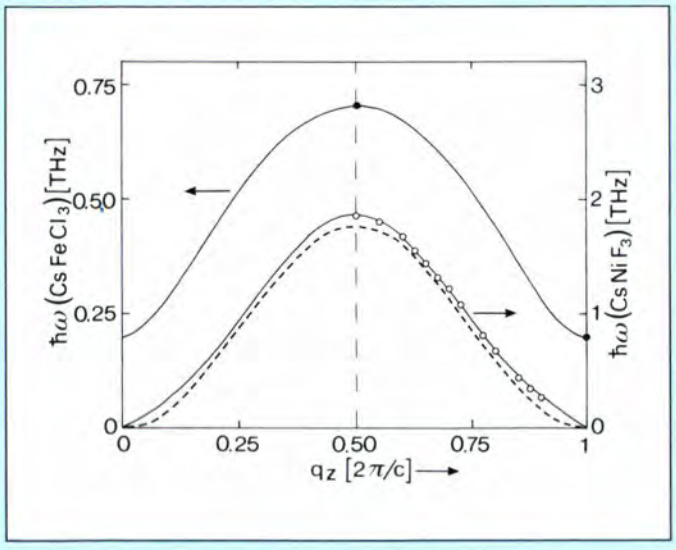


*Figure 31 Dispersion curves of the magnetic excitations in quasi-1D materials with J > 0; ● for $CsFeCl_3$ [9] eq. (9) with J = 0.063 THz and A = 0.507 THz (most of the experimental data had to be left out because they had been measured at positions where the interaction between the chains contributes); ○ for $CsNiF_3$ [5] eq.(5) with J = 0.25 THz and A = 0.106 THz (the energy scale is adjusted by the ratio $J(CsFeCl_3)/J(CsNiF_3)$ = 0.25); --- Heisenberg system eq. (2) with J = -0.063 THz (left scale) or J = 0.25 (right scale) and A = 0.*

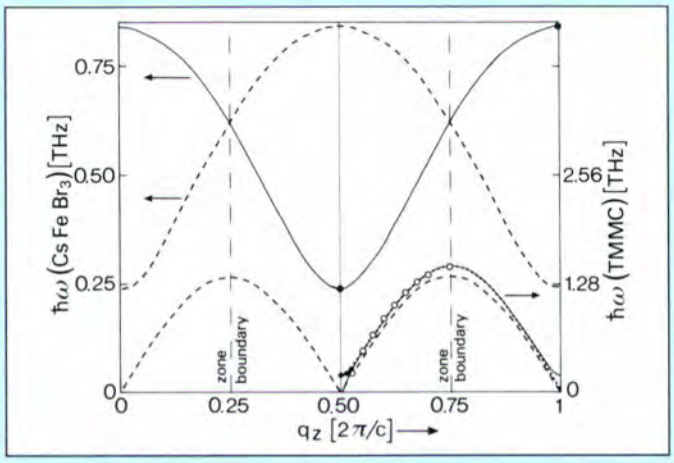


*Figure 32 Dispersion curves of magnetic excitations in quasi-1D materials with J < 0, ● and full lines for $CsFeBr_3$ [10] eq. (9) with J = 0.066 THz and A = 0.62 THz, the dashed line gives the observed mirror mode (most of the experimental data had to be left out because they had been measured at positions where the interaction between the chains contributes); ○ and full line IP-mode in TMMC [7] eq. (6) with J.S = -0.337 THz and A = 0.005 THz, and the dotted curve give the OP-mode eq. (7) (the energy scale is adjusted by the ratio $J(CsFeBr_3)/J.S$ (TMMC) = 0.195); --- Heisenberg system with J = -0.066 THz (left side scale) or J.S = -0.337 THz (right side scale) and A = 0.*

This ratio changes for $q_z \rightarrow 0$ in the way that the OP component goes to zero. The Heisenberg character of the IP component allows the frequency to go to zero for $q_z \rightarrow 0$. The best studied example is $CsNiF_3$ [5]. Fig. 31 gives the magnon dispersion curve together with the curve for A = 0 and J as for $CsNiF_3$.

**If J is negative** in an x-y system, the dispersion of the spin waves is given by [6]

$$\hbar\omega = 2S\,[(2J - 2J\cos(cq_z + \pi))\,(2J + 2J\cos(cq_z + \pi) + A)]^{1/2} \quad (6)$$

for the IP components and by

$$\hbar\omega = 2S\,[(2J - 2J\cos(cq_z)(2J + 2J\cos(cq_z) + A)]^{1/2} \qquad (7)$$

for the OP components. To understand this we have to look to the eigenvectors. First we create a new set of basic vectors $\mathbf{e}_1'$ and $\mathbf{e}_2'$ for the case A = 0.

$$\mathbf{e}_1' = \mathbf{e}_1 + \mathbf{e}_2$$
$$\mathbf{e}_2' = \mathbf{e}_1 - \mathbf{e}_2 \qquad (8)$$

As seen in Fig. 30 $\mathbf{e}_1'$ is a linearly polarized amplitude in the plane and $\mathbf{e}_2'$ is polarized out of plane. **For A > 0 the degeneracy will be lifted.** The mode $\mathbf{e}_1'$ stays Heisenberg like and extrapolates to zero frequency for $q_z = 0$, while mode $\mathbf{e}_2'$ goes to a finite frequency. This energy gap increases with increasing A. Fig. 32 gives calculated dispersion curves for TMMC (7) from eqs. (6) and (7) together with the calculated one for A = 0 and J as for TMMC.

In Fig. 32 the spinwave vector $q_z$ starts at the origin of reciprocal space goes through the Brillouin zone boundary (001/4) to the centre of the antiferromagnetic Brillouin zone (001/2) and continues through the Brillouin zone boundary at (003/4) to the next zone centre at (001). The dispersion curves in the Heisenberg and the Ising system (not drawn) are horizontal at the zone boundary and symmetric relative to it. The experimental intensity of magnons for these two systems is strong near (001/2) and vanishing near (001). The dispersion curves for the x-y system have finite but opposite slope at the zone boundary. This is known from phonon dispersion curves in non-symmorphic space groups. In TMMC [7] the mode $\mathbf{e}_1'$ has been observed near (001/2) and the mode $\mathbf{e}_2'$ with the gap around (hh1/2) with $h \neq 0$. This reference to TMMC has to be taken with some care because it has a spin = 5/2 and not 1. In $RbFeBr_3$ [8] (spin = 1) the dispersion curve has been measured along the path from (001/2) to (001). The intensity was continuous from the low frequency branch near (001/2) to the high frequency branch near (001). The high frequency mode near (hh1/2) and the low frequency mode near (hh1) was not searched for.

**For $8|J| < A$** we have a SGS system with no magnetic moment. Here we cannot any more talk about spinwaves. The excitations are excitons. It is quite interesting that now the dispersion curves are given by the same formula in both cases of positive and negative J [3], (Figs. 31 and 32).

$$\hbar\omega(q_z) = [A^2 - 8A\ J\cos(cq_z)]^{1/2} \qquad (9)$$

**For $J > 0$,** it is easily understood that the frequency has the lowest value at (000) and the highest at the zone boundary (001/2) corresponding to the Fe ion periodicity. An example for $J > 0$ in a SGS system is $CsFeCl_3$ [9], (Fig. 31).

**The case $J < 0$** is more difficult to understand because eq. (9) describes the system as if the unit cell in real space contains only one "magnetic" ion. As these ions do not carry a magnetic moment in the groundstate, they are indistinguishable. The minimum of frequency appears at (001/2) and the maximum at (001). This resembles the case of $RbFeBr_3$ [8] the x-y system with $J < 0$ as explained before. The frequency of an x-y system extrapolates to zero at (001/2), where the SGS system with $J < 0$ shows a gap. There seems to be a logic evolution from the Heisenberg system $A = 0$ with two degenerate modes extrapolating to zero at (001/2). For $0 < A < 8/J/$ the degeneracy is lifted, one mode extrapolates to zero and the other shows a gap increasing with A. For large A but still in the limit of the x-y system this gap has a higher frequency than the zone boundary mode.

Finally for $A > 8/J/$ both modes show a gap. Unfortunately so far there is **no theory which continuously describes this evolution.** The main reason is that **the x-y and the SGS system are different phases.** The experimental investigation of the SGS system $CsFeBr_3$ with $J < 0$ /10/ revealed the dispersion curve as given by eq. (9) visible all along from (001/2) to (001) where the high frequency was measured. The observation of the **mirrored mode** with low frequency at (hh1) and high frequency at (hh1/2) was no surprise for the experimentalists but it cannot be explained by theory.

The visibility of the modes in $CsFeBr_3$ can be reproduced by models $\mathbf{e}''_1$ and $\mathbf{e}''_2$ for the precession of the magnetic moments in the excited state. These eigenvectors contain necessarily antiferro- as well as ferro-magnetic configurations of the fluctuations, (Fig. 30). The elliptic shape is a consequence of the anisotropy in z-direction. $\mathbf{e}''_1$ belongs to the low frequency mode and $\mathbf{e}''_2$ to high frequency one. The dispersion curve which is predicted by eq. (9) is observed due to the IP components while the mirrored mode gets its intensity from the OP components.

**To summarize** we should like to come back to what we said in the introduction, where exchange interactions were neglected. A negative A corresponds to a magnetic groundstate in the crystal electric field splitting scheme, while a positive A corresponds to a nonmagnetic SGS lowest level. In the latter case, it is possible that the exchange interactions are strong enough, see eq. (4), to produce a phase transformation from the SGS to a planar Heisenberg system [11,12]. The $Fe^{2+}$ ion in the family $AFeX_3$ (with A = Rb and Cs; X = Cl and Br) finds itself locally in a positive anisotropy with a nonmagnetic SGS. In the Rb-compounds ($RbFeCl_3$ [13]) the exchange interaction (the one along the chains together with the one between the chains) overcomes the anisotropy and creates a magnetic groundstate, while the Cs compounds stay nonmagnetic.

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# Crystal and Magnetic Structures

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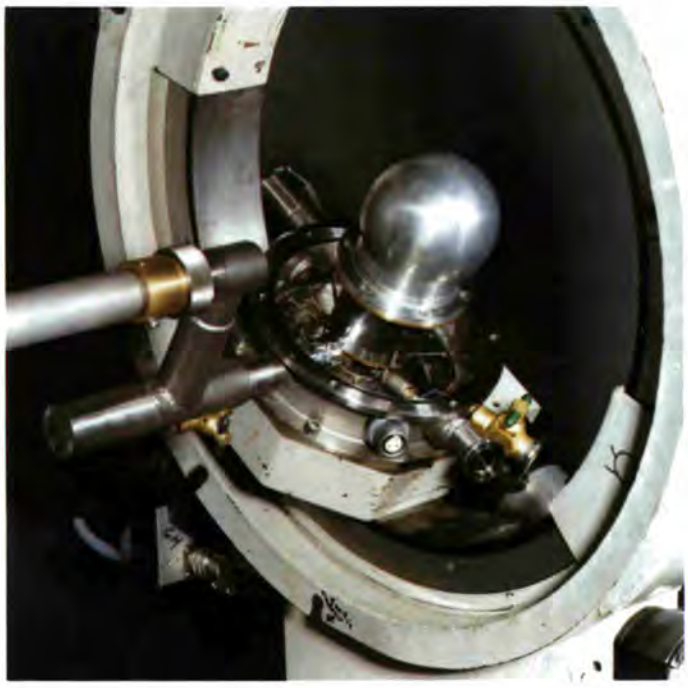


*He cryostat for the Eulerian cradle of D10*

## General Summary

1988 was an excellent year for the College with most instruments running tight schedules. The hot source single crystal instruments D3 and D9 and the powder diffractometers D2B, D1B and D1A were heavily over-subscribed as was the triple-axis spectrometer with polarization analysis, IN20.

The departure of scientists such as K. Ziebeck, W. Kuhs, and of an unusually large number of thesis students and the death of S. Wilson were sorely felt. Fortunately there is a new wave of Spanish scientists and thesis students.

Interest in high $T_c$ superconductors generated many new experiments. Although the quick access procedure again functioned well for exceptionally exciting new materials, the Scientific Council examined very critically more speculative or survey-type proposals, even those from experienced groups, and insisted on adequate sample characterization.

The demand for polarized neutron beams is far from satisfied by D3 and IN20. There is also an increasing demand for an instrument devoted to materials science and engineering science, which D1B and D1A can only partly satisfy, particularly since D1A has come back into the normal scheduling system. The third area where new solutions are being examined actively is that of position-sensitive detectors. Several instruments function routinely with p.s.d.'s, but improved versions are needed on certain diffractometers. Other instruments need completely new types of p.s.d., for example with resolution better than 1 mm, and covering much larger solid angles.

There was a significant component of diffraction work in the papers submitted to the very successful ICNS'88 (International

Conference on Neutron Scattering) held in Grenoble in July 1988, an opportunity to assess work done at the ILL in the light of outstanding contributions from most world centres.

The College produced the Autumn 1988 Neutron Diffraction Newsletter of the International Union of Crystallography, devoted to ILL diffraction instruments.

# Scientific Highlights in 1988

## Crystallography of Non-Magnetic Systems

### Alpha-uranium charge density wave

The complex charge-density-wave state appearing in α-uranium (α-U) below 43 K, discovered in 1979, has been re-examined with systematic scans (this time using the 3-axis configuration of D10 with the analyser to improve signal/noise). A new set of superlattice reflections has been observed considerably smaller in amplitude (by about a factor of 50 on average) than those of the primary set of wave vectors $\langle\vec{q}\rangle$ (= $[\pm q_x, \pm q_y, \pm q_z]$). The new satellite reflections result from a two-dimensional modulation (d = 2); a linear combination of two wave vectors in $\langle \vec{q} \rangle$ describes the satellites by the choice of two wave vectors ($\vec{q}_1 = [1/2, q_y, q_z]$ with $\vec{q}_2 = [-1/2, -q_y, q_z]$).

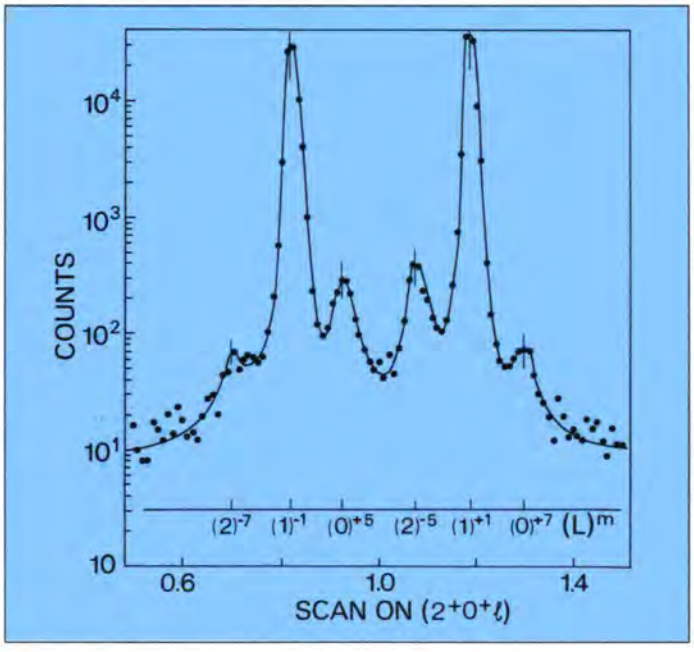


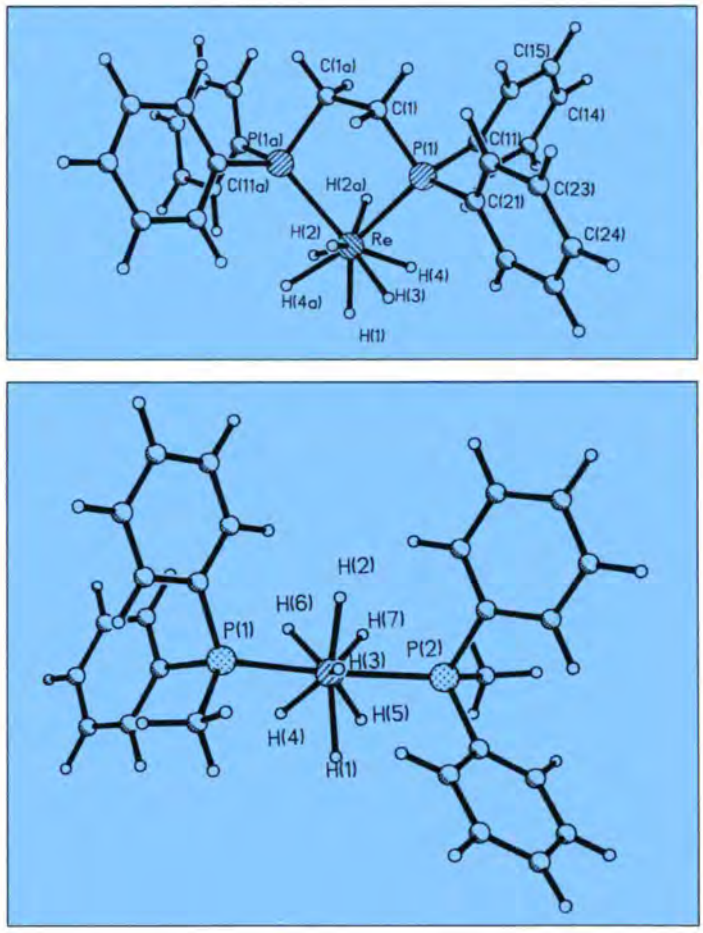

*Figure 33 Intensities of the mth-order satellites in the diffraction pattern of α-U as a function of l along the line (2.5, 0.176, l ) at T = 15 K, and indexing up to the mth-order ($m = p_1+p_2$) using pairs of modulation wave vectors $\vec{q}_1$ with $\vec{q}_2$ (see text); convention for the inset: $l = L + mq_z \equiv (L)^m$.*

The result of the indexing is given in Fig. 33 where the mixed high-order satellites of the diffraction pattern are labelled using:

$$\vec{h} = h\vec{a}^* + k\vec{b}^* + l\vec{c}^* = H\vec{a}^* + K\vec{b}^* + L\vec{c}^* + \sum_{n=1}^{d} \rho_n \vec{q}_n$$

(H,K,L: Miller indices).

The choice of wavevectors $\vec{q}_1$ and $\vec{q}_2$ sets special constraints on the super space group of the charge-density wave state, a subject that has recently been of theoretical interest. The present analysis aims at a more detailed determination of the wave form of the distortion in α-U (Collaboration with CNRS, Grenoble and EITU, Karlsruhe).




*Figures 34 & 35*
*Classical geometry of the core hydrides in two Re(VII) complexes as revealed by single-crystal neutron diffraction.*

### Classical Re(VII) polyhydrides

The accepted formulation of rhenium polyhydrides e.g $[ReH_7(PR_3)_2]$, as complexes of Re(VII) with seven discrete hydride ligands, has been questioned in the literature with suggestions that $\{ReH_5(H_2)(PR_3)_2\}$ may be more correct, thereby reducing the oxidation state of Re to (V). The present evidence for coordinated molecular dihydrogen, in this series, has come entirely from proton n.m.r relaxation parameters ($T_1$) and not from any structural studies.

During this year, two members of this important family of complexes, have been studied on D19. There was slim evidence from low temperature X-ray data in only one of them for the seven core hydride positions, and these were in fact ambiguous with an indication for the ($\eta$- $H_2$) moiety. The basic polyhedral geometry in these high coordination number systems is known to vary with the nature of the phosphine ligand, and therefore this, as well as the vital core hydride ligation mode, was studied by single-crystal neutron diffraction.

With the chelating diphosphine, dppe (1,2 bis-(diphenylphosphine) ethane), crystals were grown as a tetrahydrofuran solvate of high sensitivity, while crystals where ($PR_3$) = ($PPh_2Me$) were more robust. Both of these complexes have now been shown unequivocally to adopt the classical configuration in the solid state, at T = 200 K, for (dppe); and 15 K for ($PPh_2Me$). (Figs. 34 and 35), with no H....H contacts between the seven core ligands shorter than 1.6 Å. The application of D19 to this type of chemical problem is thus well illustrated (Collaboration with Universities of Bristol and Salford).

## Single-crystal reflection integration on line

Two-dimensional position sensitive detectors are now in routine use for the collection of single crystal Bragg reflection data for structure determination on the diffractometers D19, D9, D17 and DB21. A further detector is shortly to become available on D15. While position sensitive detectors in principle give the ability to measure the intensities of reflections more precisely than traditional monodetectors, they also present the challenge of how to deal quickly and on line with the large amounts of data. In the case of D19, for example, 12-30 Mbytes of data are collected per hour in the course of a typical experiment and this must be processed in real time if the progress of the experiment is to be directed satisfactorily.

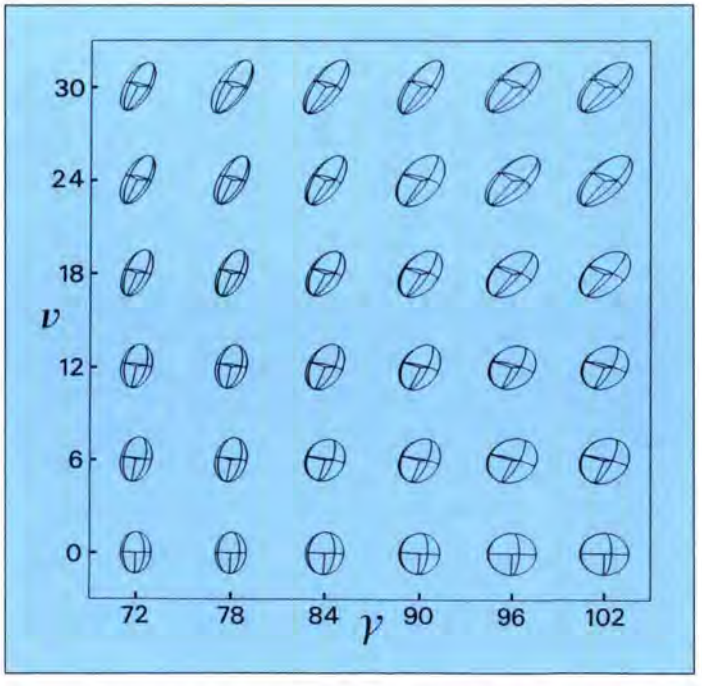



*Figure 36 Library of reflection shapes observed at high scattering angles on D19. The ellipsoid projection has $\gamma$ horizontal and $\nu$ vertical.*

The data from two-dimensional detectors can be represented as a three-dimensional array of integers in "detector space", built from "frames" of data on which the events over a given time at each spatial element of the detector are registered. Successive frames represent crystal rotation steps and Bragg reflections occur at predictable points in this distorted reciprocal space. The task of the data processing program is to integrate as accurately and precisely as possible the intensity associated with each individual Bragg reflection. When the reflections are strong and the background is low, this presents little problem and a simple "shoe box" integration technique is adequate. When the reflections are weak and sit on a high background, however, the task is more difficult and a more sophisticated method must be used.

An algorithm based on the $\sigma(I)/I$ method of Lehmann and Larsen for monodetector scans has been developed for quickly and accurately integrating Bragg reflections in three-dimensions. Using *a priori* knowledge from strong reflections (in the form of their shapes and integrated intensity profiles), the algorithm significantly improves the precision with which the intensities of weak reflections can be measured and compares favourably with that which can be achieved by a least squares profile fit. The ellipsoidal shapes of the 95% integrated intensity contours for strong Bragg reflections collected in an experiment on a Re(VII) heptahydride complex on D19 are shown in the Fig. 36 as a function of the $\gamma$ and $\nu$ angles measured on the diffractometer. The smooth variation in shape is also present in the weak reflections, and it is this knowledge which allows their intensities to be more accurately integrated.

In common with other *a priori* methods, the derived intensity is subject to systematic error if the *a priori* information is incorrect. In the case of the $\sigma(I)/I$ method, most errors bias the intensities negatively. One obvious source of error is a significant difference between the predicted centre of a weak reflection (where the integration volume is centred) and its actual centre. This emphasizes the need for a good UB matrix for the description of the peak positions and care must be taken to make the minimum acceptable integration volume sufficiently large to allow for errors of this type. Having collected three-dimensional data, however, does offer the possibility of recovery from having a poor UB matrix at experimental measurement time, as this can be refined later from the observed centres of strong reflections.

A second problem common to all integration algorithms is how to define the background. This is especially difficult in the presence of strong thermal diffuse scattering when the peaks have long tails which can give rise to a high background estimate and a negative bias to the derived intensities. The definition adopted with this algorithm is that a local background is measured in an ellipsoidal shell immediately around the peak. This appears to give internally consistent results; the merging R factors on the intensities of symmetry related reflections do compare favourably with the uncertainties predicted by the algorithm and for D19 are typically about 3% (Collaboration with Kings College, London).

## Stroboscopic measurements on silicon

In the last few years much progress has been made in the field of high-speed time-resolved neutron measurements for the observation of processes in the microsecond range. To perform these measurements a stroboscopic method is used. The method is applicable to all cyclic processes, where data collection in a short time window (slice) of length T(3) can be repeated over a large number of synchronized cycles n. Assuming that in a given time interval T(3) of a synchro-cycle the system under investigation is seen by neutrons in exactly the same physical state, the data collected at a constant delay time T(2) over a huge number of cycles can be added. By varying the delay T(2) the total evolution in time of the observed phenomenon can be reconstructed plotting the signal (e.g. a Bragg peak) against the delay time. At the moment the data can be stored in a maximum of 160 slices per 128 channels, each channel corresponding to one of the 128 cells of the D20 position sensitive detector. The minimum length of the time window is T(3) = 2 μs with a minimum distance of 2500 μs between two slices.

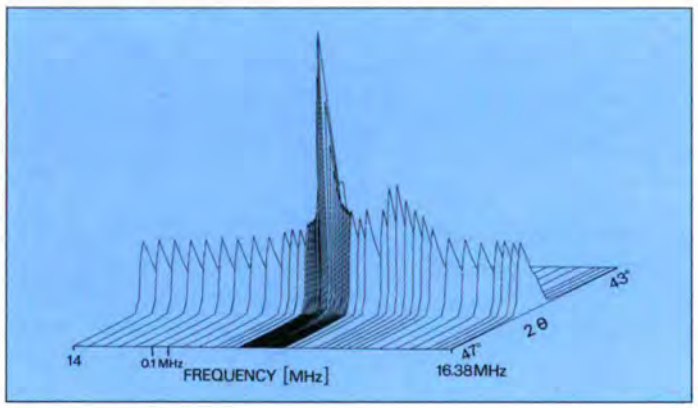



*Figure 37 Frequency response of a vibrating Si crystal.*

The stroboscopic method demands for certain experiments specifications matched by D20: a high flux (~2 x $10^7$ $cm^{-2}s^{-1}$) to collect data in a reasonable number of cycles and a multidetector to observe a whole Bragg peak or even the complete powder pattern simultaneously. Currently the time resolution is about 50 μs for 2.4 Å neutrons, and about 15 μs for a neutron wavelength of 0.8 Å.

In a recent experiment done on D20, we studied the time response of a silicon single crystal to the application of a longitudinal sound wave. For this D20 was used in its 4-circle mode in connection with the stroboscopic measurement unit. Sound was excited by means of a 15 MHz x-cut quartz transducer glued onto one side of the plate-like silicon crystal.

The crystal was set up in Bragg geometry. In a first part of the experiment the frequency response of the coupled transducer - silicon sandwich was measured by tracing the **111** peak intensity as a function of applied rf-frequency. Fig. 37 shows the results obtained. The resonance frequency shows up as a maximum reduction of extinction at 15.21 MHz. A second intensity maximum is seen at 15.63 MHz caused probably by a second parasitic resonant mode of the transducer-silicon system. Such a reduction of extinction in vibrating crystals is a well known phenomenon.

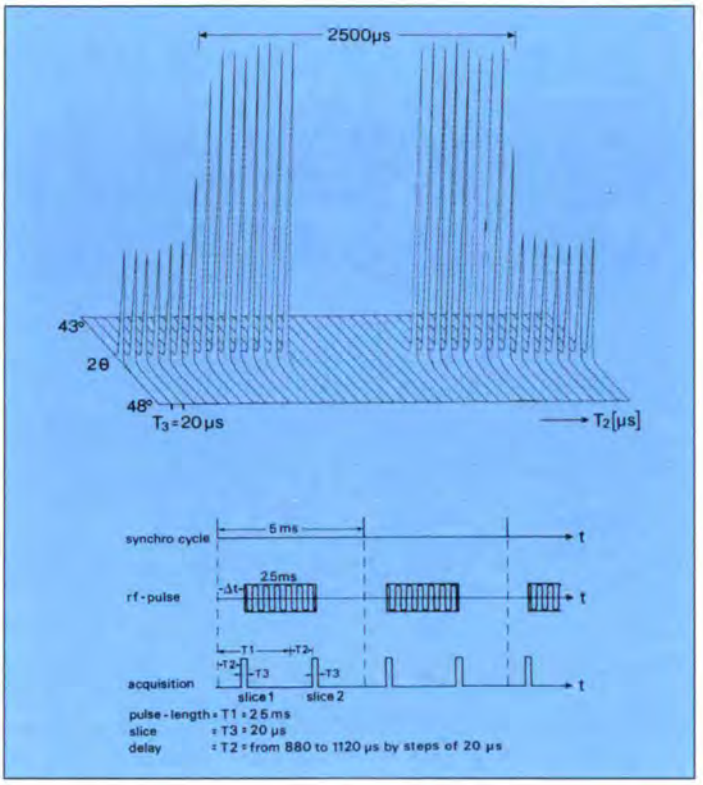



*Figure 38 Time-response of a Si crystal to a pulsed ultrasonic field.*

In a second part of the experiment we were interested in the time response of the crystal to an applied pulsed sound field. The quartz transducer was excited with an rf-pulse of 2500 μs duration and a repetition rate of 5000 μs corresponding to one synchro-cycle.

In this case the stroboscopic unit was used such that two slices of length T(3) = 20 μs separated by T(1) = 2500 μs were shifted over the transition "non-vibrating/vibrating crystal" by the use of a variable delay time T(3). The total counting time for a peak was t = 0.5 s corresponding to n = 25000 cycles. Fig. 38 (top) shows the silicon **111** peak against the delay time. In Fig. 38 (bottom) we present schematically the time position of the applied rf-pulse as well as the two time slices shifting over the ultrasonic pulse. The response of the crystal to the sound excitation is almost instantaneous. Only one intermediate intensity value between the vibrating and non-vibrating state is seen. We conclude that the rise time of the Si **111** peak due to the pure reduction of extinction by vibrating the sample is smaller than the current time resolution of the experimental set up. The result is in agreement with the fact that the transducer should vibrate resonantly after approximately 3 rf-frequency periods corresponding to 3 μs at 15 MHz. The method therefore allows for a precise measurement of the time resolution of the detector.

The experiment was a first test of the time resolution achievable on D20 for stroboscopic measurements. This information is needed for further experiments and for the interpretation of a recent experiment on a vibrating magnetic erbium iron garnet crystal at low temperature done on D9. Here we observed what we interpret as a sound induced spin

reorientation at about 15 K. Without the agitation of a sound wave polarised along the (**111**) direction of the sample the reorientation takes place at temperatures between 60 K and 80K. In interpreting the results obtained one has to be extremely careful, because the application of the sound field also heats the sample; nor can pure domain effects be excluded.

Here time resolved measurements could help to distinguish between instantaneous sound induced effects, heating and domain relaxation, because of the different characteristic time behaviour.

## Deformation of granitic rocks

The deformation behaviour of polyphase rocks has generally been approached by assuming that the material is homogeneous and can be described with a macroscopic flow law. However, microscopic processes in rocks which are composed of harder and softer crystals of different grain shapes are bound to be more complicated. A first step toward understanding these processes, was to investigate texture development of granodiorite which has been progressively deformed to mylonite and phyllonnite in the Santa Rosa mylonite zone in Southern California. Chemical and mineralogical composition do not change during this transformation.

It is extremely difficult to measure pole figures of materials with complex diffraction patterns; it was necessary to use sophisticated neutron diffraction techniques with high resolution and position-sensitive detectors which enables mathematically deconvoluting at least part of the spectrum. We were able, using data measured on D1B, to construct separate pole figures for biotite, quartz and plagioclase (Fig. 39). Some thousands of powder patterns were deconvoluted almost in real time with the automatic option of ABFfit (1987 Annual Report). In granodiorite preferred orientation is weak. In mylonite a regular fabric of biotite and quartz develops, whereas feldspars remain randomly oriented. Quartz has c-axes in the "a" fabric direction as is common in mylonites. Interestingly, biotite a-axes show preferred orientation, suggesting that slip may be involved. In phyllonite with a grain size of about 50 μm, the biotite fabric is very strong. The quartz fabric has largely vanished, perhaps by superplastic processes which do not produce texture. A weak but regular plagioclase fabric develops (Collaboration with University of California, Berkeley).

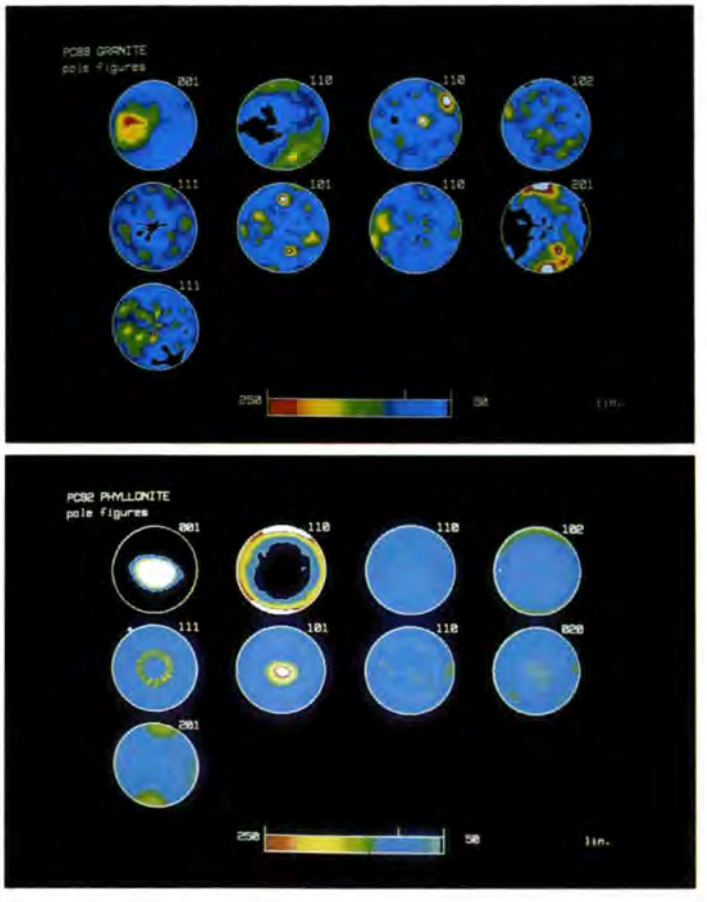



*Figure 39 Pole figures for granite and phyllonite from data measured on D1B.*

## Crystallography of Magnetic Systems

### Polarized neutron powder diffraction on Laves phases compounds

When studying the magnetic properties of cubic Laves phases $RT_2$ where R represents a rare earth and T a transition metal, the common opinion was that the magnetic moment seen in susceptibility measurements stems only from the transition metal atom. $YFe_2$ and $ZrFe_2$ were thought to be simple ferromagnets. Systematic studies on mixed phases $A(Fe_{1-x}B_x)_2$ where A = Y, Zr, Gd, Ho and B = Mn, Co, U were undertaken to study the magnetism of the 3d-electrons of Fe.

Only recently, however, band structure calculation cast doubt on the simple ferromagnetic picture; a ferrimagnetic arrangement seemed to be preferred. D1B with its newly developed option of polarized neutrons was the ideal instrument to check this idea. Measuring in neutron spin up and spin down configuration the difference plot $I\uparrow - I\downarrow$ (Fig. 40) showed immediately the existence of a moment on the Y-site orientated antiparallel to the Fe moment (the

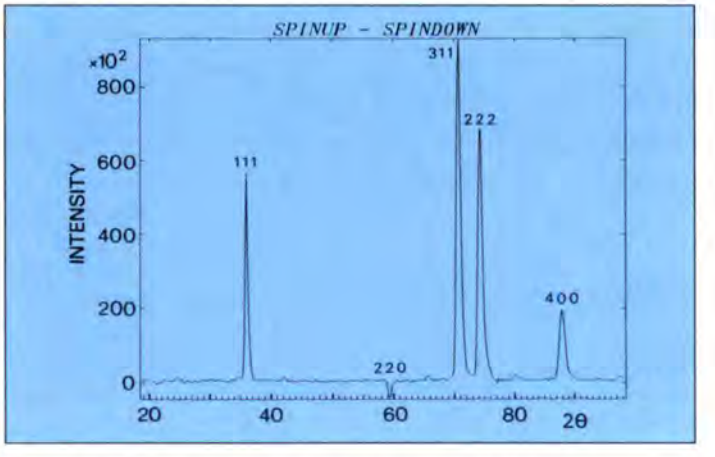



*Figure 40 Spinup-spindown powder spectrum of $YFe_2$.*

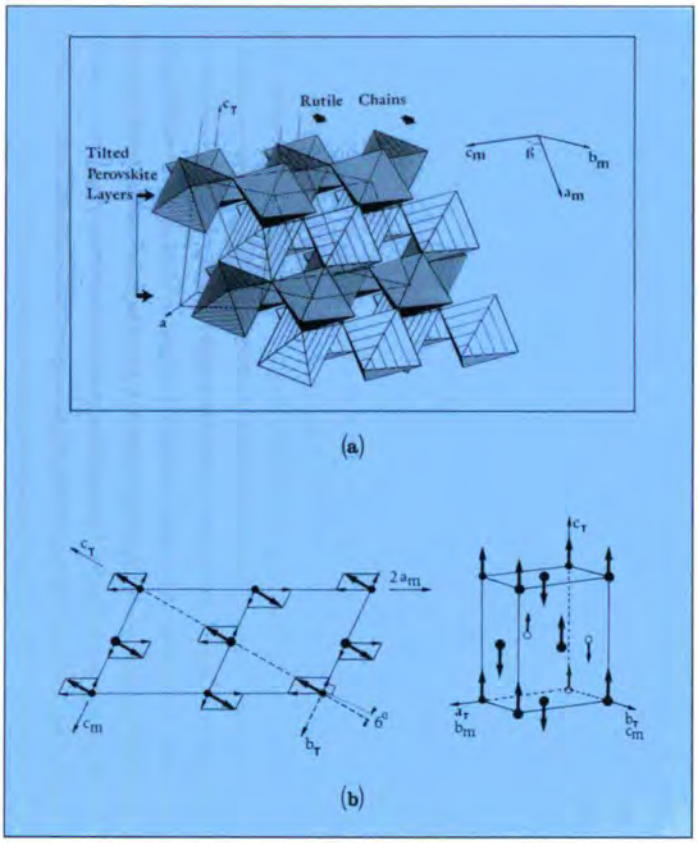



*Figure 41* *(a) Crystal structure of the dirutile $LiCoF_4$, in terms of co-ordination polyhedra. The view summarizes the two possible descriptions of this structure. [$CoF_6$] octahedra are heavily hatched.*
*(b) Two projections of the magnetic structure of $LiCoF_4$. The magnetic moments are perpendicular to the perovskite planes.*

structure factor for the 220 reflection arises only from the Y-site). Knowing the depolarization of the beam as it traverses the incompletely magnetized sample the flipping ratios I↑/I↓ for the first 5 reflections allowed the extraction of the magnetic moments. Values of $\mu Fe = 1.77 \pm 0.08\ \mu_B$ and $\mu Y = 0.56 \pm 0.04\ \mu_B$ were found. This result, simple as it is, will surely stimulate the rediscussion of a whole series of experiments on the afore mentioned mixed phases.

## Crystal and magnetic structures of $ACoF_4$ (A = Li, Cs)

Recently, two new cobalt (III) fluorides with original structures have been synthesized: $LiCoF_4$ and $CsCoF_4$. $LiCoF_4$ and the isostructural $LiMnF_4$ were the first compound shown to exhibit the dirutile structural type. $CsCoF_4$, isostructural to β–$RbAlF_4$, provides a striking illustration of how magnetic frustration is solved in an Ising-like antiferromagnet (due to strong single-ion anisotropy of the $3d^6$ $Co^{3+}$ cations). The crystal and magnetic structures of both compounds were refined from powder diffraction patterns collected on D1B and D1A (collaboration with the University of Giessen).

The neutron diffraction study of $LiCoF_4$ confirmed the dirutile structure of this compound. The $Li^+$ and $Co^{3+}$ cations alternate along the chains of edge-sharing octahedra of the structure, resulting in the doubling of the **c** axis compared to a simple rutile cell. This structure can also be described as the stacking of tilted perovskite layers of [$CoF_6$] octahedra between which $Li^+$ cations are located (Fig. 41a). The magnetic structure implies a doubling of the **a** axis of the monoclinic cell. $LiCoF_4$ is a collinear antiferromagnet, with magnetic moments perpendicular to the perovskite planes (Fig. 41b).

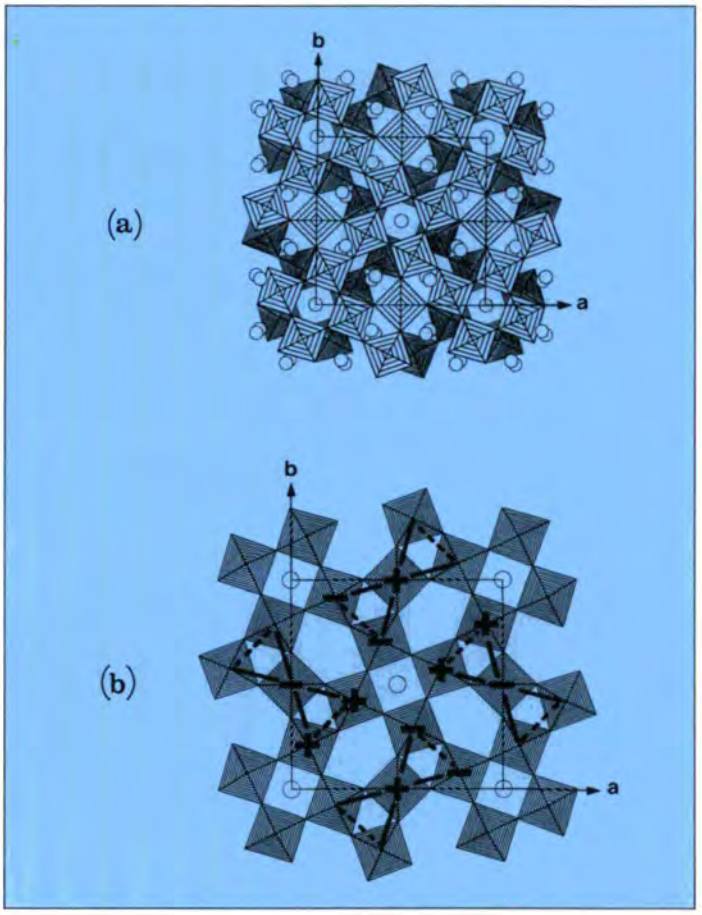



*Figure 42* *(a) Crystal structure of $CsCoF_4$, projected along the **c** axis. Two successive tetragonal bronze-type layers (octahedra with different hatching rates) are drawn. They are isolated from each other by $Cs^+$ cations (circles).*
*(b) Magnetic structure of $CsCoF_4$. The magnetic moments are aligned along the **c** axis of the structure. Frustrated triangular plaquettes are underlined; broken lines represent broken antiferromagnetic interactions.*

The structure of $CsCoF_4$ is built up from disconnected layers of the tetragonal tungsten bronze structural type (Fig. 42a). In this structure, all super-exchange angles Co-F-Co are larger than in $LiCoF_4$, giving evidence for the antiferromagnetic character of magnetic interactions in $CsCoF_4$. The presence of odd cycles of antiferromagnetic interactions leads to magnetic frustration, but the strong anisotropy of $Co^{3+}$ ions prevents the magnetic moments from adopting a non-collinear three-sublattice arrangement, as in a frustrated Heisenberg antiferromagnet.

The competition between interactions in triangular plaquettes is solved by the breaking of the weakest interaction, that is the one with the smallest super-exchange angle (Fig. 42b).

## Magnetism of rare-earth disilicides

Rare-earth disilicides have recently received considerable attention due to the possibility of obtaining epitaxial layers on silicon, which may lead to important technological applications. The crystallographic and magnetic structures of some of them have been studied using the D1B diffractometer. The crystallographic structure (orthorhombic Imma) was confirmed for $NdSi_{1.8}$, $DySi_{1.75}$ and $HoSi_{1.7}$ (phases in equilibrium with silicon). Néel points occur at 10, 10.5 and 14 K respectively. $NdSi_{1.8}$ is a simple antiferromagnet below 6 K with a moment $M_x$ = 1.68 $\mu_B$ parallel to the propagation vector Q = 1/2, 0, 0). $HoSi_{1.7}$ is a canted antiferromagnet, with two inequivalent propagation vectors $Q_1$ = (1/2, 0, 0) and $Q_2$ = (0, 1/2, 0), which may arise from different domains.

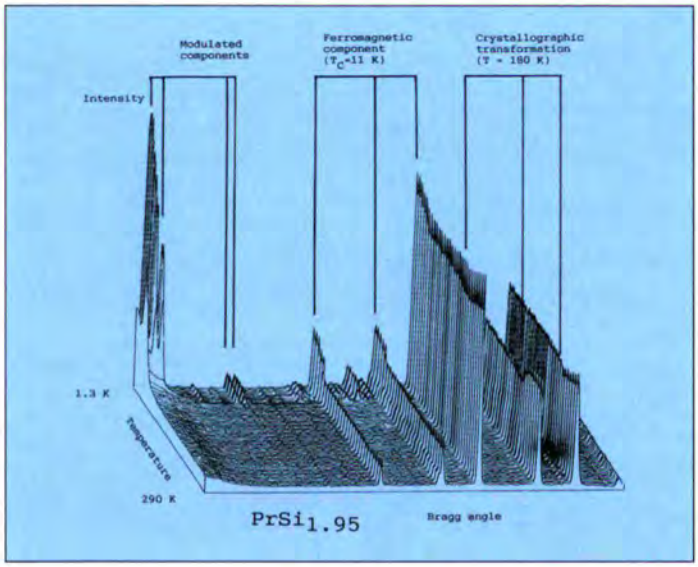



*Figure 43 Thermo-diffractogram of $PrSi_{1.95}$ between 1.3 and 290 K, obtained on D1B ($\lambda$ = 2.52 Å) for the 2$\theta$ range $2° < 2\theta < 82°$.*

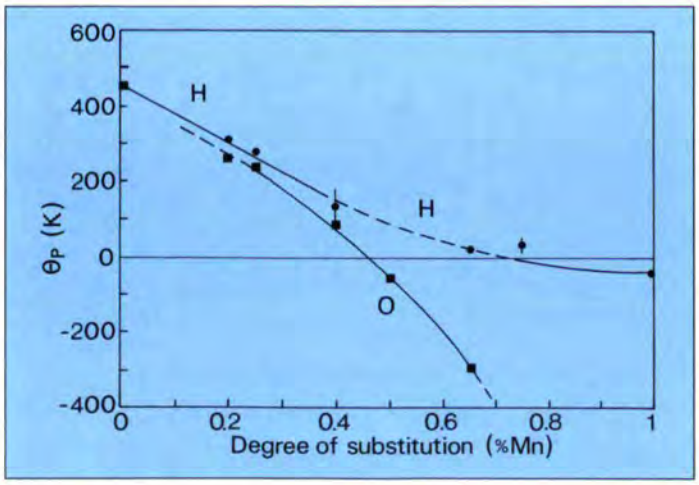



*Figure 44 Evolution of the paramagnetic Curie temperature $\theta_p$ as a function of the substitution rate and also of the crystallographic phase. The point with x = 0.4 in the H phase has a large uncertainty due to the difficulty of obtaining a single phase by quenching at high temperature.*

Some structural ambiguities remain, for instance either of the modes $F_yA_x$ or $F_yA_z$ may be associated with $Q_1$ = (1/2, 0, 0). The largest component (7 $\mu_B$) lies in the a, b plane. $DySi_{1.75}$ has a complex structure: the $M_x$ component (5.2 $\mu_B$) is associated with $Q_1$ = (0, 0, 0), whereas the $M_z$ component is modulated, being associated with vectors $Q_2$ = (1/2, 1/2, 0) and $Q_3$ (1/2, 1/2, $\tau$ = 0.14).

A possible explanation for this situation may be a more or less periodic modulation of the crystalline electric field from site to site, which in turn may be related to the distribution of the Si vacancies. In the case of $PrSi_{1.95}$ a crystallographic transformation occurs near 180 K from a tetragonal ($I4_1/amd$) phase to another (slightly distorted) phase, both co-existing at lower temperature. Magnetic ordering occurs at 11 K, one phase being ferromagnetic, the other being a modulated phase with a long period (Fig. 43).

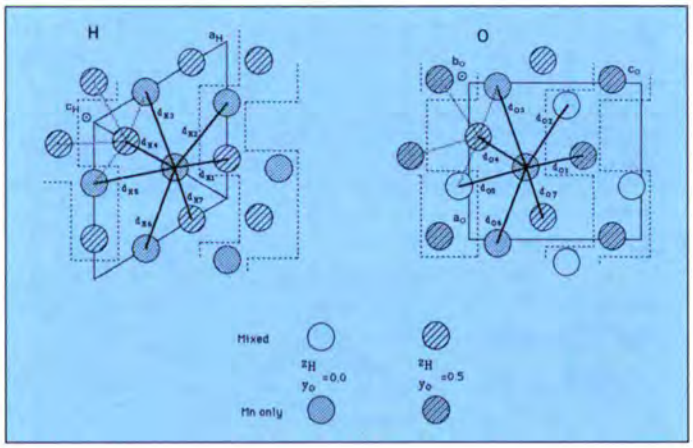



*Figure 45 Comparison of the metallic environments for a composition with $x \geq 0.5$. The dotted circles represent the positions occupied by Mn atoms only. Dashed lines define the areas where the crystal transformation modifies the populations. Calculation of the interatomic distances shows that the most affected distances are those related to modifications in the populations:*
*$d_{H1}$ = 2.76 Å, $d_{O1}$ = 2.81 Å*
*$d_{H2}$ = 3.14 Å, $d_{O2}$ = 2.65 Å*
*$d_{H5}$ = 3.14 Å, $d_{O5}$ = 2.82 Å*

## Influence of crystal structure changes on magnetic couplings in $(Fe_{1-x}Mn_x)_2P$

Compounds of the system $(Fe_{1-x}Mn_x)_2P$ crystallise in two forms depending on the value of x and the thermal treatment. Some intermediate compositions may also be obtained in either hexagonal (H, space group $P\bar{6}2m$) or orthorhombic (O, s. g. Pnma) phases. Both of these phases are those generally encountered in the MM'X series (M, M': transition metals, X: As or P) in which metal atoms are distributed within two kinds of coordination polyhedra formed by phosphorus: pyramidal and tetrahedral. In $Fe_2P$ ferromagnetic couplings are predominant. Evolution of the paramagnetic Curie temperature ($\theta_p$) as a function of the degree of substitution indicates an enhancement of the antiferromagnetic couplings as the proportion of Mn increases (Fig. 44). In order to connect this

evolution with the relative importance of the amount of manganese and the intermetallic distances, a neutron diffraction study of the crystal structures was undertaken on powder samples. From the evolution of $\theta_p$, compositions x = 0.20 and x = 0.65 were thought most appropriate to study the influence of these parameters. The difference in the scattering length of iron and manganese allows the metal ordering to be accurately determined and the preferential occupation of the pyramidal sites by the manganese atoms was confirmed. The difference in the metal environments are indicated in Fig. 44. There are two types of environments depending on whether the metal is located in the pyramidal (10 neighbours) or the tetrahedral site (8 neighbours). Calculation of interatomic distances shows that shortest Mn-Mn distances (i. e. $d_{Mn\text{-}Mn} < 3.45$ Å) are close to those found in α-Mn. In α-Mn the exchange integral is negative for $d_{Mn\text{-}Mn} \leq 2.8$ Å (antiferromagnetic) and changes sign for larger distances. Variations of x from 0.2 to 0.65 does not involve distance variations greater than about 0.1 Å in the O phase. The degree of substitution appears then as the predominant parameter. An increase in x corresponds in the O phase to an increase in the number of Mn-Mn bondings whose lengths range between 2.6 and 2.8 Å. Correspondingly the strength of the overall antiferromagnetic couplings in the O phase is much greater.

From Fig. 45 it is seen that the distances most affected by the structure change are those related to a change of atom type. The other distances vary by less than 0.15 Å. Taking account of the altered distances and the modifications of the atom type, the difference in the exchange energies corresponding to the (H) and (O) phase can be expressed as:

$$\Delta J = J_{Ex}(H) - J_{Ex}(O) = 2J_{Mn\text{-}Mn}(3.13\ \text{Å}) - \beta\, J_{Mn\text{-}Mn}(2.65\ \text{Å})$$

with β the proportion of manganese on the mixed positions.

The most important term concerns $J_{Mn\text{-}Mn}(3.1$ Å) which is positive and tends to reduce the antiferromagnetic couplings. The larger the value of β the larger is the overall difference ΔJ, in agreement with the experimental data on $\theta_p$.

The evolution of the Curie temperature is therefore closely related to the degree of Fe-Mn substitution and in the case of a particular composition, with the variations of the interatomic distances between the nearest metallic neighbours. In $(Fe_{1-x}Mn_x)_2P$ the change of crystal structure (O → H) is associated with a change in the proportion of manganese atoms linked together by short bonds.

## The dynamic form factor of Invar

In all models of the thermal expansion anomaly known as the Invar effect it it recognized that the relationship between magnetic moment and atomic volume or interatomic distance plays a crucial role. Hence the interactions between the vibrational and magnetic degrees of freedom are of great importance. One very sensitive way to study such interactions is through the polarisation dependence of the magneto-vibrational scattering which probes the extent to which the magnetization density around individual atoms depends upon thermal displacements. In a recent experiment, carried out on IN20 the polarisation dependence of the neutron cross-section in two longitudinal acoustic modes of $Ni_{.65}Fe_{.35}$ at 100K has been measured. The data have been

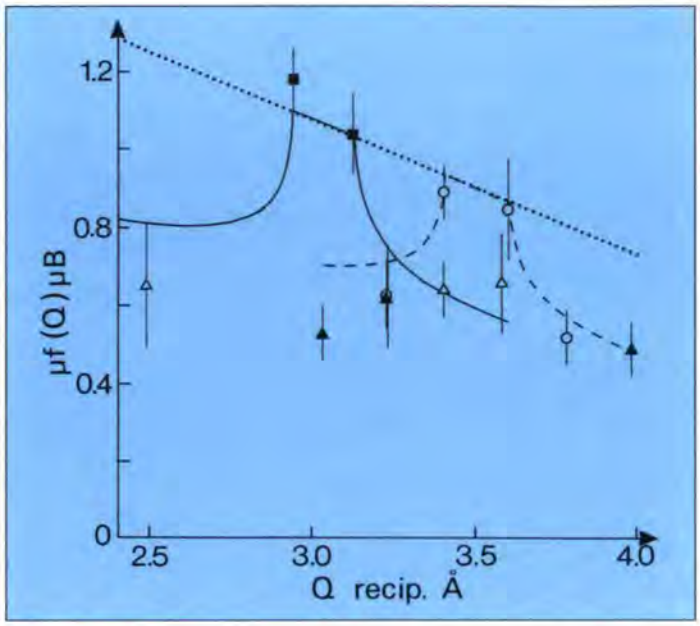



*Figure 46 Experimental values of the product μf(Q) plotted against Q. The points marked with squares and triangles are for the LA 111 phonon at $K_f$ = 2.66 and 4.1 Å$^{-1}$ respectively; those marked with circles and crosses are similar for the LA 001 phonon measured from 002. The dotted line indicates the moment variation due to the form factor f(Q) and the full and dashed curves show the predictions of the simple model described in the text for the LA 111 and LA 001 modes respectively.*

analysed to give the ratio between the magnetic and nuclear cross-sections for magneto-vibrational scattering. Assuming that this ratio is $(f(Q)\mu/\bar{b})^2$ where f(Q) is the form factor, μ the mean magnetic moment, and $\bar{b}$ the mean nuclear scattering length, the product f(Q)μ was evaluated and is plotted against Q in Fig. 46.

It can be seen from the figure that the product f(Q)μ varies much more rapidly with Q than does the static form factor f(Q). Indeed it is not a monotonic function of Q but appears to decrease with increasing energy transfer as q increases on either side of the Bragg peak for both phonon branches that were measured. As yet a fully quantitative treatment of this behaviour cannot be given, but qualitatively it shows that the moment μ contributing to the magneto-vibrational scattering at high q and E is reduced by a factor of about two with respect to the mean periodic moment. The full and dashed curves correspond to a crude model in which the moment drops to zero for that part of the phonon vibration in which the near neighbour distance is reduced from its equilibrium value by a critical amount which is about 0.03 Å for the curves shown.

## Neutron and synchrotron topography: two complementary techniques for the observation of first order magnetic phase coexistence

Neutron and synchrotron radiation topography have proved to be valuable, and complementary, techniques for the investigation of the coexistence of two magnetic phases. These

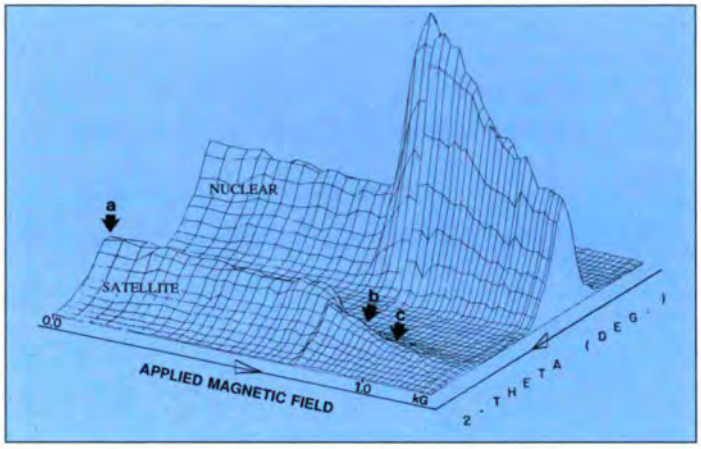



*Figure 47 Rocking-curves in neutron diffraction for the 200 reflection and one of its satellites, when the phase transition is induced by magnetic field.*

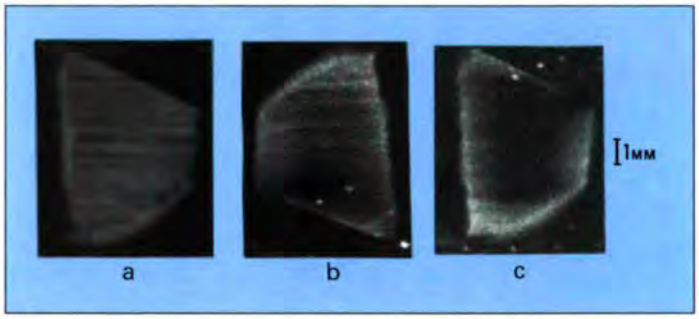



*Figure 48 Neutron topographs made at 32 K using the magnetic 2+δ, 0, 0 satellite in applied field (a) 0, (b) 0.088 T, (c) 0.098 T.*

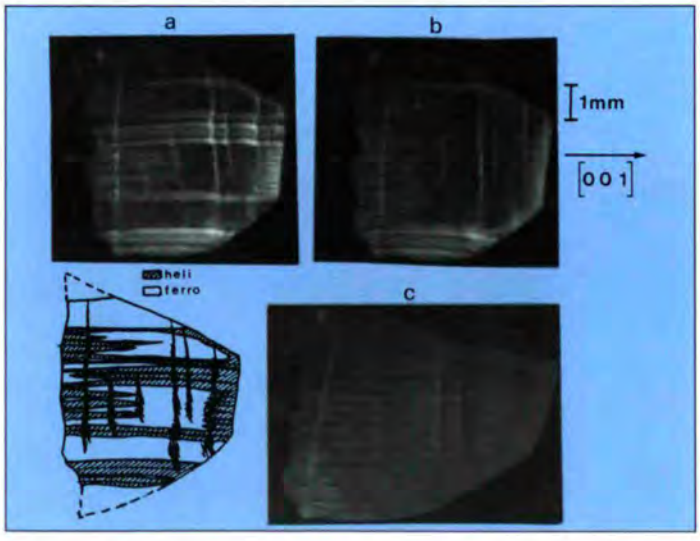



*Figure 49 Synchrotron radiation (white-beam) X-ray topographs. Temperature 15 K. Applied field (a) 0.213 T, (b) 0.216 T, (c) 0.189 T.*

first order phase transitions are driven by several energy terms (magnetostatic, elastic, interface) which can be in conflict. This results in a rich variety of situations which have been only scarcely investigated. In the past experiments were mainly performed by optical methods on transparent samples. With neutrons and synchrotron radiation the observation of the shape and evolution of the interface can be extended to non transparent samples but their main contribution resides elsewhere. Neutron diffraction topography leads to the unambiguous identification of the magnetic phases. Synchrotron topography, on the other hand, helps to understand the inhomogeneous distortions associated with the interfaces.

An investigation performed on a highly perfect crystal of MnP constitutes an example of this type of approach. This compound exhibits an antiferromagnetic helical phase below $T_S \approx 47$ K, and is ferromagnetic between this temperature and $T_C \approx 291$ K. Below 47 K small magnetic fields (< 3 KGauss) induce the ferromagnetic phase. The observation of the phase coexistence was performed by varying the magnetic field. Severe extinction effects are observed in both phases, but are reduced during the phase coexistence, leading to a dramatic increase of the intensity of the nuclear peak (Fig. 47). Chirality domains are visible on a topograph performed on a magnetic satellite reflection at zero field (Fig. 48a). This effect corresponds to a domain-related reduction of extinction. Figs. 48b to 49c give information about the phase coexistence. The interfaces are mostly parallel to both the applied field and the magnetization (Figs. 48b and 49c), the helimagnetic region being rejected to the edges of the sample at the end of the transition (Figs. 48c and 49b). This pattern minimizes the predominant magnetostatic energy term, but not the elastic one. Synchrotron topographs (recorded at LURE) indicate the inhomogeneous distortions which arise during the transition. A spectacular example is the enhancement of the image size (Fig. 49c) which occurs when the field is decreased starting from the ferromagnetic phase. This effect is associated with an overall curvature of the sample .

The same helimagnetic-ferromagnetic transition occurs very differently in the temperature driven case; here the magnetostatic energy term is much reduced by the presence of domains in the ferromagnetic phase, and other energy terms (elastic, interface) mainly drive the phase coexistence.

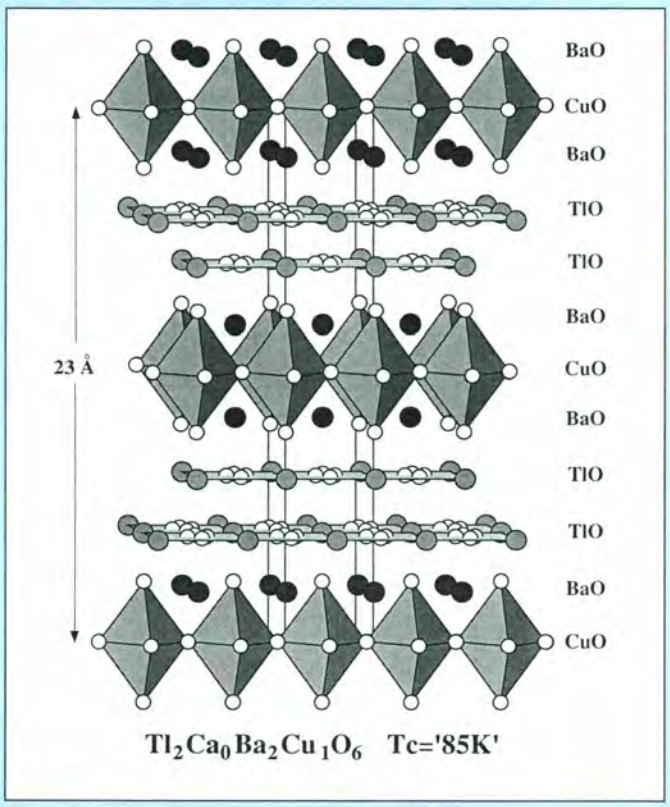
Secretaries: S. Mason

J. Baruchel

## High Tc Superconductors and Powder Diffraction

A. W. Hewat

The contribution of neutron powder diffraction to understanding the structures of the new high Tc superconductors was widely recognized in 1987, with the ILL-CNRS work of Capponi et al. [1] being one of the eight most cited papers in all of science for the 12 months to June 1988. Many new superconductors were discovered in 1988, and the ILL was quick to continue the competition in this area, largely in collaboration with the CNRS Grenoble crystallography laboratory, but also with other laboratories in France, Britain, Germany and the USA (Figs. 50, 51 and 52).




*Figures 50 to 52*
*The structures of three thallium superconductors studied at the ILL;*
*$Tl_2Ca_0Ba_2Cu_1O_6$, $T_c$ = 85 K.*

In January, very precise measurements of the low temperature variation of the structure of $YBa_2Cu_3O_7$ were completed by a Geneva-CNRS-ILL collaboration [2]. The orthorhombic distortion, produced by the CuO-chains along the b-axis, was found to show subtle changes, especially near Tc in some samples. The strong disorder of oxygen within these chains was found to decrease with temperature, indicating dynamic disorder, but appeared constant below Tc as if the disorder became static. Later, a Tübingen-ILL group extended these measurements to high temperatures, showing how this chain disorder resulted in oxygen mobility [3].

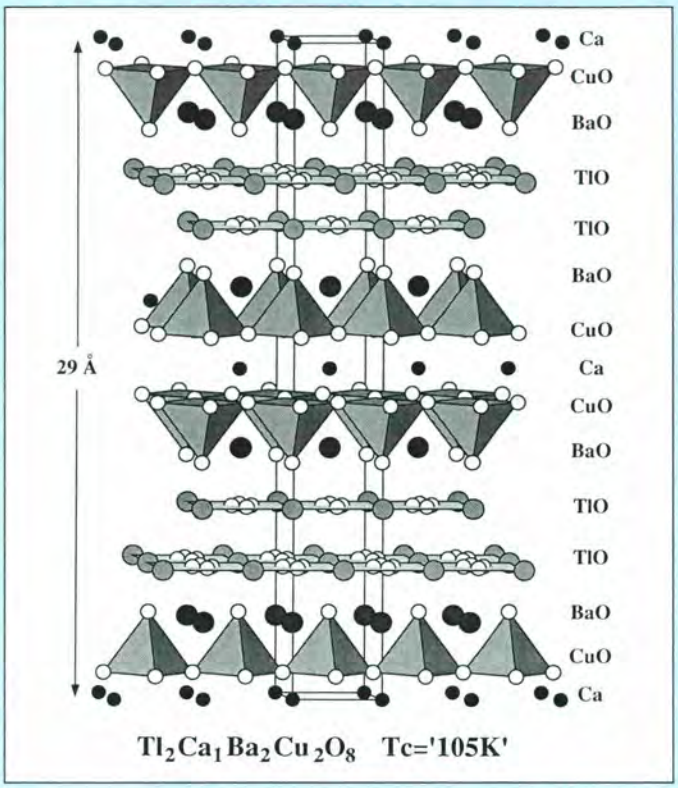



*Figure 51 $Tl_2Ca_1Ba_2Cu_2O_8$, $T_c$ = 105 K.*

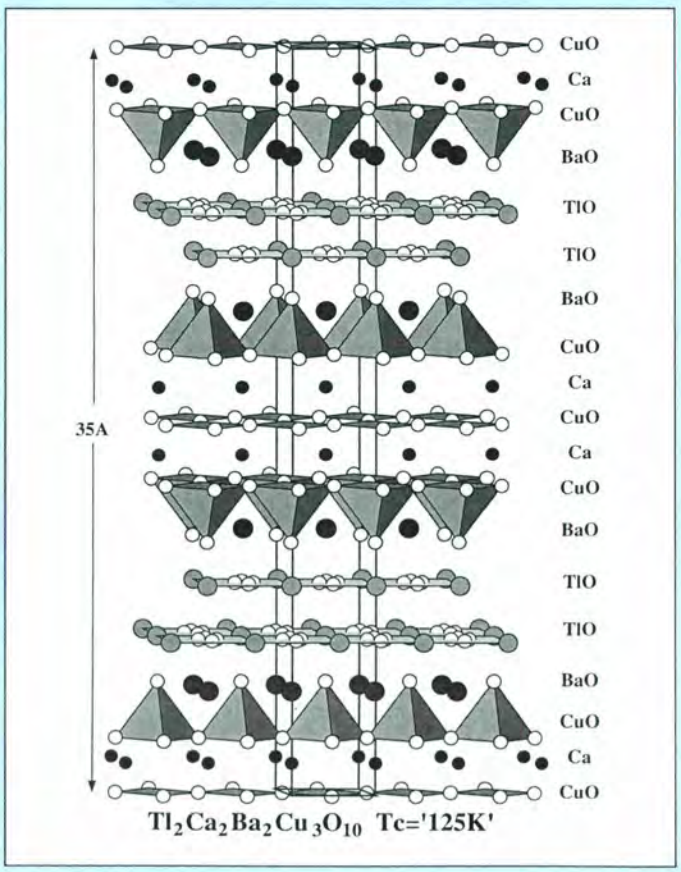



*Figure 52 $Tl_2Ca_2Ba_2Cu_3O_{10}$, $T_c$ = 125 K.*

At the same time, a Spanish-ILL group made a complementary type of measurement on the temperature dependence of the structure of $YBa_2Cu_3O_{7-\partial}$. Using the rapid data collection possibilities of D1B, they studied the dynamics of the loss of oxygen from the structure on heating, and the uptake of oxygen on cooling [4]. Their results (Fig. 53) demonstrate the great sensitivity of neutrons to small changes in the oxygen stoichiometry, and are important for understanding the best methods of fabrication of such materials, as well as their fundamental properties.

An Oxford group was meanwhile continuing work on D2B on the original $La_2CuO_4$ superconductor doped with various cations, and with various oxygen stoichiometries. They were primarily interested in the possible magnetic properties of the material, but finished by obtaining very precise measurements of the low temperature variation of the tilts of the $CuO_4$ octahedra, and their relation to structural phase transitions [5]. These structural questions are attracting renewed interest as the importance of magnetism for superconductivity begins to appear less plausible.

In February, the structure of the new bismuth material $Bi_2Ca_1Sr_2Cu_2O_8$ was refined from a pure sample from the

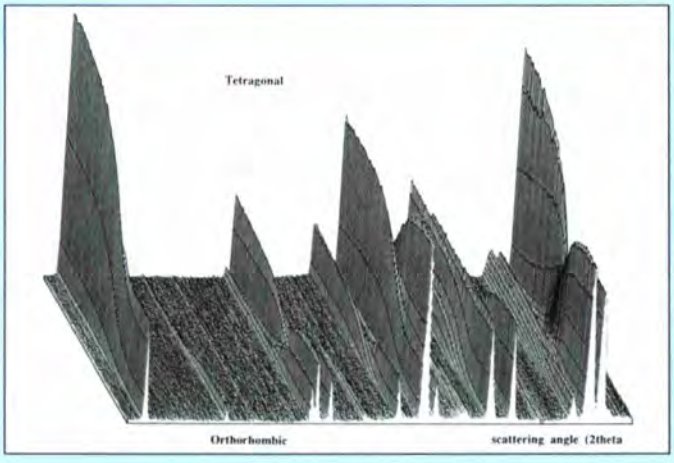



*Figure 53 Kinetics of the orthorhombic-tetragonal transition in $YBa_2Cu_3O_{7-\partial}$ with oxygen loss.*

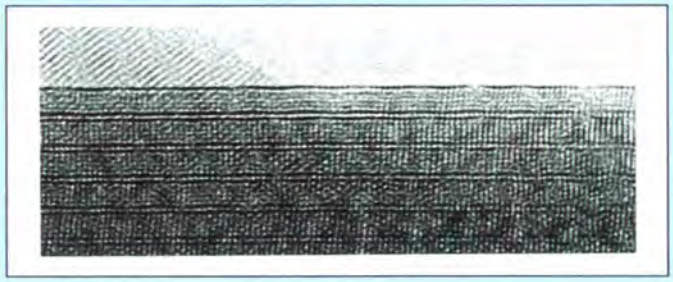


*Figure 55 High resolution electron microscope image of $Bi_2Ca_1Sr_2Cu_2O_8$ showing that the structure cleaves between BiO planes, supporting the neutron result that there is no Bi-O-Bi interplanar bonding.*

CNRS. The oxygen atoms were located, and the bismuth coordination and apparent valence determined, resolving conflicting X-ray results from DuPont and Bell-IBM laboratories. The X-ray results could not agree whether oxygen was between the BiO-layers, as in the classical 'Aurivillius' structure, or whether the oxygen was instead within the layers. The neutron work showed that the latter model was correct. This was important for understanding the valence state of bismuth. This work was then extended to give a detailed model for order-disorder of oxygen within the BiO-planes, which was later found to be similar in the thallium isomorphs. Fig. 54a shows the disordered model first found [6], with a pseudo-tetragonal structure, and Fig. 54b, the final ordered model with local orthorhombic symmetry [7].

This neutron work was complemented by high resolution electron images of the structure (Fig.55) which showed that the material cleaves between BiO-layers, confirming the absence of inter-layer Bi-O-Bi bonding [8]. The electron diffraction also helped understand the 'incommensurable' superstructure observed in this material.

A similar situation was found in $Tl_2Ca_1Ba_2Cu_2O_8$, though the superstructure is less marked, and a better refinement of the average structure is then possible. A Warwick- ILL-Oak Ridge team obtained a rather precise structure (Fig. 51b) from an exceptionally pure single phase sample [9]. Apart from confirming that the Tl coordination was similar to that for Bi, with an interesting difference because of the absence on Tl of

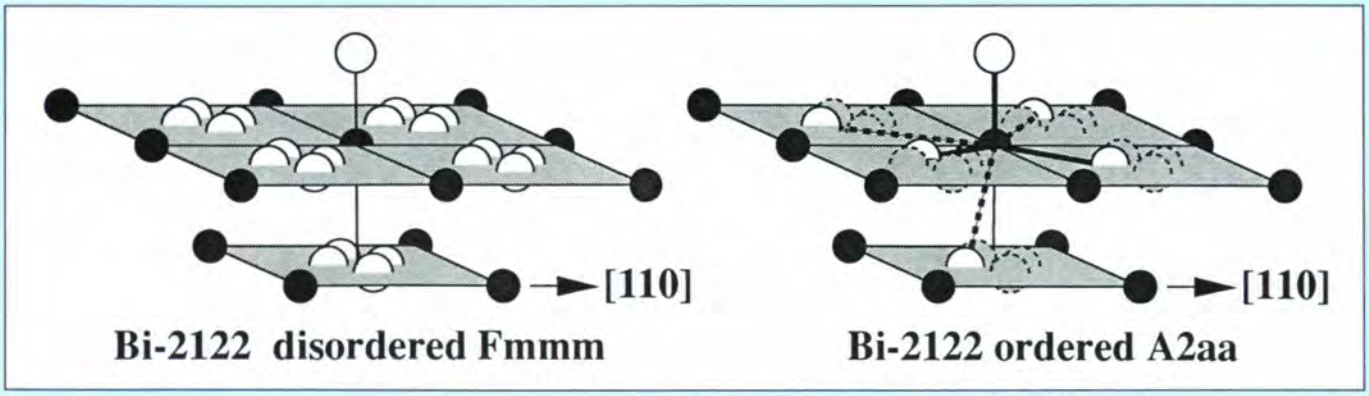



*Figure 54 Models for the BiO-layers in $Bi_2Ca_1Sr_2Cu_2O_8$ showing a) disorder of oxygen in a pseudo-tetragonal superconducting phase, and b) ordered oxygen displacements to produce microscopic orthorhombic symmetry.*

the Bi lone pair electrons which keep the BiO-planes apart, this work was important for showing that the stoichiometry of the material was close to that of the nominal formula; only a few percent of atoms were missing in the TlO plane. It had been proposed that much larger deviations from stoichiometry were necessary to produce the electron hole concentration apparently necessary for high Tc.

The most interesting phase is no doubt $Tl_2Ca_0Ba_2Cu_1O_6$ (Fig. 50a), which had been reported with a wide variety of transition temperatures. A CNRS-DRF-ILL team investigated a large number of different samples, some superconducting and some not [10]. It had been supposed that the difference might be due to a difference in stoichiometry, but this work showed that it was instead a difference in *symmetry*, or rather in *atomic order*. Well ordered samples were found to be orthorhombic and non-superconducting, while disordered samples were found to be pseudo-tetragonal and superconducting, as for the higher members of this series of compounds. By order is meant both order of the superstructure and order of the oxygen atoms within the TlO planes. Since the effective cation valence is reflected by the oxygen coordination and distances, disorder may indicate valence fluctuations.

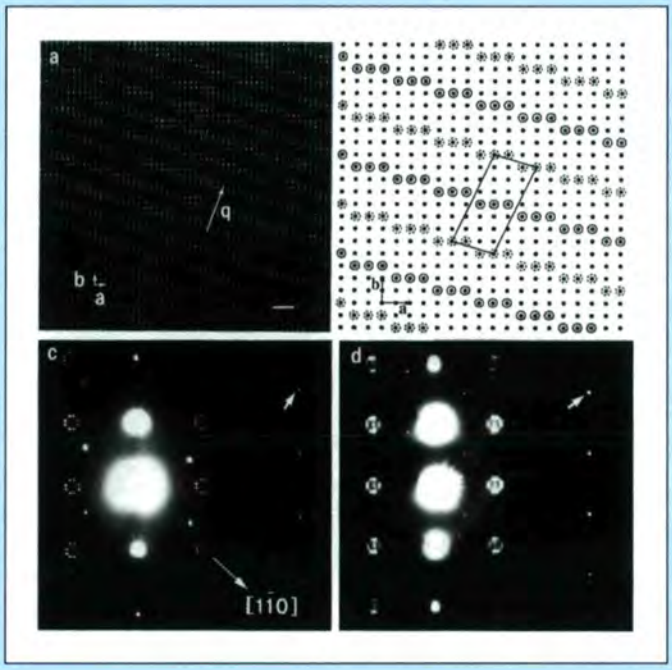



*Figure 56 Electron image*
*(a) of the superstructure in $Tl_2Ca_0Ba_2Cu_1O_6$, which can be explained by a model,*
*(b) in which 1 in 8 TlO atoms are missing, as required by neutron diffraction. The non-superconducting orthorhombic modification shows long range order, with sharp superlattice spots,*
*(c) while there is only very short range superlattice order in the superconducting pseudo-tetragonal modification, where the spots are very diffuse,*
*(d) as in the higher order phases.*

In $Tl_2Ca_0Ba_2Cu_1O_6$ neutron diffraction showed that as many as 1 in 8 of the atoms in the TlO plane were missing, and that in well ordered samples the superstructure was particularly evident. In fact the superstructure could be imaged at the atomic level (Fig. 56a), and by systematically removing every 1 in 8 thallium atoms a model was obtained (Fig. 56b) to correspond with this electron microscope image [11].

The superstructure is seen to be exceptionally sharp in the non-superconducting orthorhombic samples (Fig. 56c), indicating long range order, while in the superconducting pseudo-tetragonal samples (Fig. 56d), the superstructure is as diffuse as it is for the higher order phases.

What then is the effect of adding more copper oxide layers? The second member of the series $Tl_2Ca_1Ba_2Cu_2O_8$, with two copper oxide layers always appears to be 'tetragonal' and superconducting, with a higher Tc. With three copper oxide layers $Tl_2Ca_2Ba_2Cu_3O_{10}$ has the highest Tc = 125 K that can be reproduced reliably. It has been proposed that adding more copper oxide layers increases the electron hole concentration, and thus increases Tc, so that Tc should increase indefinitely.

We therefore investigated the oxygen co-ordination and cation valence in this third phase. The result was that the third copper oxide layer, which unlike the others has no oxygen outside the CuO-plane, appeared from the bond lengths to have no extra electron holes; all of the copper appeared to be $Cu^{++}$! Perhaps the additional copper oxide layer merely stabilizes the 'tetragonal' structure needed for high Tc [12]. In that case, adding even more copper oxide layers should not much increase Tc. This indeed appears to be the case.

High temperature superconductivity is a good test of the validity of the ILL system of neutron beam use. It has been said that the proposal system means that no exciting new experiments can be done, but superconductivity has shown this to be false. The ILL is widely recognized as being a leader in this area, as indicated by the number of invited papers at specialized international meetings, and the US citation index. The proposal system has worked well for the programs of measurement on well characterized materials, but the ILL has proved sufficiently flexible that it has also been possible to achieve a number of 'firsts' on new materials in competition with much larger materials laboratories. No one in the member countries can claim that they have had an exciting new sample and have been denied rapid access to ILL facilities. Of course some of the experiments were also unsuccessful, but this is in the nature of the new science.

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*The International Conference on Neutron Scattering was held from 12 to 15 July 1988 at the Grenoble University Campus. It was jointly organized by the CEN Grenoble and the ILL (about 450 participants attended).*

## Real Time Crystallography

J. Brown

Real time crystallography is the name given to crystallographic studies of non-equilibrium systems undergoing chemical or physical changes during the course of an experiment. Experiments in real time crystallography may be divided into two types depending on whether the change undergone is reversible or irreversible. For the study of irreversible processes such as chemical reactions a high neutron flux is essential. The time resolution of such an experiment is that needed to collect a set of diffraction data of sufficient precision to characterize the state of the sample. It may be argued that real time studies are the domain of X-ray scattering where the very high brilliancy of synchrotron sources allows much better time resolution than can be obtained with neutrons. However for relatively slow reactions neutron scattering has the advantage of giving better contrast for changes involving light atoms and particularly those involving water. By sampling a much larger volume of material neutron scattering gives a better picture of the state of the whole sample if it is heterogeneous.

Irreversible reactions must almost always be studied with polycrystalline samples, and the ideal instrument for such experiments is a high flux powder diffractometer with a position sensitive detector covering a wide range of scattering angle. Many of the pioneering experiments in this field were carried out on the instrument D1B at ILL which has a multi-wire detector covering an angular range of 80° in scattering angle with a resolution of 0.2°. With this instrument neutron diffraction patterns may be collected on a five to ten minute time-scale and reactions which reach equilibrium within one to two hours may conveniently be studied. A good example of this type of experiment is the study of reactions between $D_2O$ and $Ca_xAl_yO_z$ by Christensen and Lehmann.[1] The reactions of the metastable calcium silicates and aluminates to produce stable hydrates are of great importance as they are the processes which occur when mixtures of cement and water solidify. They typically have reaction times between a few hours and many years. Examination of the evolution of the patterns as the process proceeds allows the identification of intermediate phases and can reveal the rate limiting steps in the reactions. A similar but simpler reaction is that of $CaSO_4.{}^1/_2\ D_2O$ with $D_2O$[2] ; the reaction involved in the setting of plaster of Paris. Fig. 57 shows the diffraction patterns observed at 307 K as a function of time. The peaks due to $CaSO_4.2D_2O$ grow and those due to $CaSO_4.{}^1/_2\ D_2O$ shrink as the reaction proceeds, however the latter decrease very rapidly whereas the former increase only rather slowly which suggests that the reaction takes place through an intermediate gel phase.

Reversible changes can be studied on a smaller time-scale than irreversible ones by applying a periodic constraint to drive the system and a stroboscopic technique to probe its response. In this way diffraction data characteristic of different stages of the cyclic process may be accumulated over many cycles. The time resolution is limited only by the response time of the detector and associated electronics and may be as low as a few microseconds. A stroboscopic detection system is available on the instrument D20 at ILL and the first real experiment carried out using it is described in the College 5 contribution to this report. In this experiment the response of a silicon crystal to a pulsed ultrasonic strain was investigated. It is known that the intensities of the Bragg reflections of silicon increase when the crystal is made to vibrate at ultrasonic frequencies. This increase is due to induced imperfection in the crystal lattice causing a reduction in extinction. By using the stroboscopic technique it was possible to show that the response to the sound excitation is almost instantaneous. This suggests that the lattice imperfection is induced directly by the ultrasonic vibration rather than by subsequent relaxation processes.

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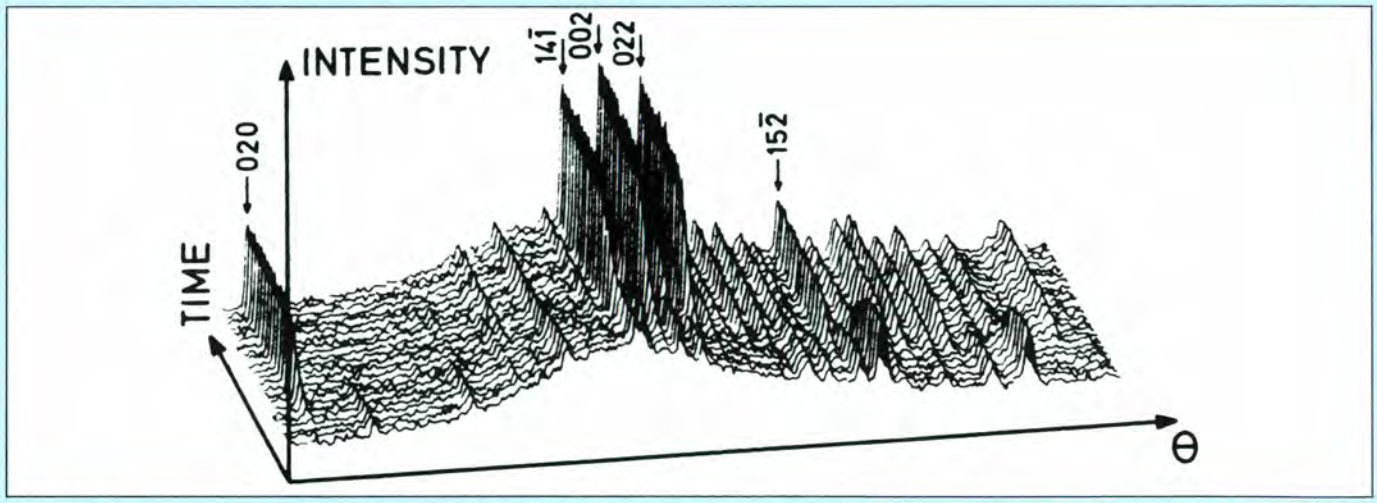


*Figure 57 The diffraction patterns at 307 K showing increasing intensities of $CaSO_4.2D_20$ and decreasing intensities of $CaSO_4.{}^1/_2\ D_20$. Each pattern in the diagram represents a time period of 5 min.*

# Liquids, Disordered Materials and Metal Physics

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## Summary

The research activities of College 6 continued successfully in 1988. A central part of these activities was highlighted during an ILL Workshop in September. Stimulating contributions belonging to the dynamics of the glass transition, low frequency excitations, spin- and orientational glasses and fractal dynamics were given. The large number of about 100 participants and the variety of the experimental methods presented - not to forget the theoretical contributions - should be mentioned. Regarding the proposed experiments the fields of main interest did not change too much with respect to previous years. The slight decrease in the number of proposals may be connected to the reduced number of reactor cycles this year.

## Scientific Trends and Highlights in 1988

### Quantum liquids

The detailed experiments on quantum liquids are continuing and have an influence on the improvement of instruments (see IN6). Recent results from measurements on **liquid $^3$He** show that the local order affects the excitation spectra (Scherm, Dianoux, Fak, Guckelsberger, Koerfer, Stirling). The particle-hole energies of $^3$He-$^4$He mixtures confirm Greywalls dispersion. The shape of the particle-hole line excitation is well described by a free Fermi gas model in range $0.25 < T/T_F < 6$.

Measurements on **liquid $^4$He** on IN12 (Stirling, Glyde, Hayden) aimed at studying the long wavelength excitations. There have been very few systematic studies in this low q regime, where the small structure factor leads to significant experimental difficulties. The experiments have shown that the $q = 0.4$ Å$^{-1}$ spectra are little affected by the change from superfluid to normal fluid behavior.

### Gases

From structural investigations of gaseous $^{36}$Ar on D4B at several low densities (n = 0.9, 1.4, 1.9, 2.4 atoms/nm$^3$) the pair potential could be extracted (Frederikze, de Graaf, van Well, van Tricht, Barocchi, Magli, Chieux). The experiment covered a q-range between 0.2 and 10 Å$^{-1}$. Special care had to be taken for the thermal insulation of the capillary inside the cryostat, connecting the sample container and the pressure control system outside in order to prevent instabilities (1%) due to condensation. Background measurements were done using $^3$He to get the correct scattering and absorption cross-sections. The extracted pair potential is shown in Fig. 58 and compared with theoretical predictions. Significant deviations are found for the low q-values. $C_o(q)$ and $C_1(q)$ are the coefficients of a virial expansion valid at low densities. $C_o(q)$ is equal to F(q), the Fourier transform of $f(r) = \exp(-U(r)/kT)-1$, where U(r) is the pair potential. $C_1(q)$ is determined by both pair potential and three body potential effects. The latter might lead to the increase of $C_1(q)$ at low q-values.

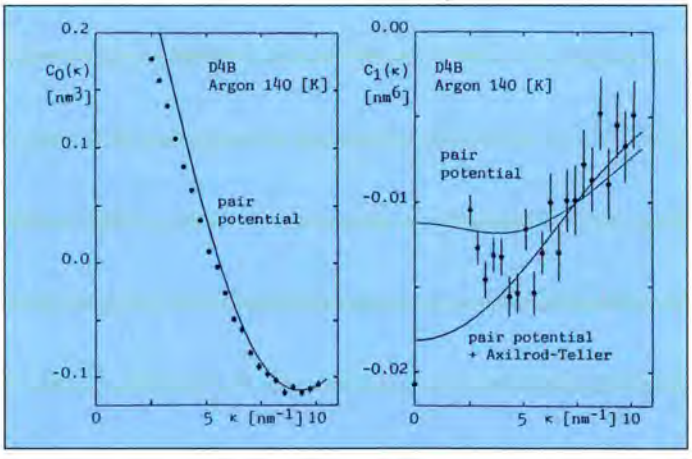



*Figure 58 Virial coefficients $C_0$ and $C_1$ compared with D4B measurements on Ar.*

### Brillouin scattering on gases

The conditions under which Brillouin scattering may be observed using neutrons impose conflicting instrumental constraints. On the one hand high energy-resolution and low momentum-transfer are required in order to observe the inelastic features - this normally implies long neutron wavelengths. On the other hand, the velocity of the neutron needs to be about 50% higher than the speed of sound in the sample, which excludes the use of long neutron-wavelengths for dense samples. As less dense samples are considered so the

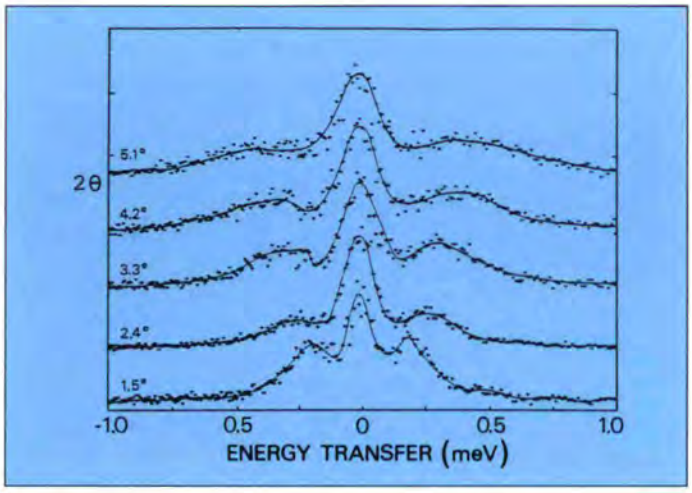



*Figure 59 Brillouin lines of gaseous $N_2$, measured on IN5.*

splitting of the inelastic peaks from the elastic peak decreases requiring the use of longer neutron wavelengths to obtain adequate energy-resolution, *vide supra*.

Despite these constraints it is possible to study favourable systems. The first experiment using existing instrumentation was carried out on compressed nitrogen gas using the time-of-flight spectrometer IN5. This spectrometer has good flux between 3.0 and 6.5 Å allowing the study of materials with sound velocities between about 1000 and 400 m/sec with energy-resolutions of 510 and 50 μeV, respectively. A Q-resolution of ca. 10% was achieved by using the spare D11 multidetector in place of the normal small-angle detectors, at a distance of 4.0 m from the sample. The detector elements were coded into 3.0 cm-wide concentric rings with 500 channel time-of-flight energy-analysis for each ring.

Some of the results are summarized in Fig. 59 corresponding to experiments using incident wavelengths of 4.7 Å with the nitrogen sample pressure of 0.51 Kbar. At higher momentum transfers the two propagating modes can be seen to move to higher energies with a marked broadening of the peaks. Other incident wavelengths and sample pressures were also explored. At a given pressure, the variation of the energy-transfers of the propagating modes with momentum transfer is found to be almost linear with a slope that agrees well with the known velocity of sound. It seems that despite the shape of the nitrogen molecule, at room temperature compressed nitrogen behaves as a hard-sphere fluid.

## Structure of liquids and amorphous materials

The isotopic substitution method has still been extensively used in order to obtain the local structure around a substituted atom.

The hydration number of the heavy lanthanide ions (M) $Dy^{3+}$ and $Yb^{3+}$ in aqueous solutions has been determined from the first order difference functions and their Fourier transform. The M-O and M-D peaks integration is unambiguously equal to eight. This conclusion is reinforced by the observation of the $r_{MO}$ distances (Merbach, Cossy, Enderby, Barnes). Further investigation is planned to obtain the coordination around the light lanthanide elements where a coordination of nine is expected.

The local environment around calcium gives a valuable insight into the local structure of oxide glasses. Although it is now clear that the local environment around strongly covalently bonded atoms like silicon or boron is almost as ordered in glasses as in crystals, there is much less information about the surroundings of "network modifying ions" like calcium. Glasses containing almost 50% CaO have been examined. The resulting correlation functions - comprising weighted sums of the Ca-centered partial functions only - give a direct representation of the first neighbour shell of oxygens. This is found to be well-defined and similar to that for crystalline $CaSiO_3$. Information on the second neighbour shell is also obtainable and the Ca-Ca first neighbour distance, inferred indirectly, does not correspond to random substitution in suitable interstices of the $SiO_2$ network structure but again is similar to that observed in the crystal (Eckersley, Gaskell, Barnes, Chieux).

Although the isomorphic substitution method has often been discarded as non reliable, there are cases, such as the substitution between Mn and the Fe-Cr mixture, which have now been proved to be really isomorphous. The structure of the liquid alloy $Al_{80}(Mn_x(FeCr)_{1-x})_{20}$ has been studied by this method and the partial structure factors have been determined (Maret, Pasturel, Senillou, Dubois, Chieux). The $S_{NN}$ function and the distribution of transition metal atom pairs, point out to the existence of a definite topological ordering. However, it is difficult to characterize clearly the type of local ordering existing in these liquid alloys.

Finally, the studies on the structural and dynamical characteristics of molecular liquids is experiencing a rapid growth. On the one hand, the basic theoretical framework has evolved rapidly, on the other hand the simulation in either the canonical or microcanonical ensembles of fluids of increasing complexity is now accessible. The liquid 1,2 dichloroethane-$d_4$ has been studied at several temperatures (Bermejo, Enciso, Dore, Chieux, Garcia, Santoro). The intramolecular parameters have been determined as well as the relative populations of *trans* and *gauche* conformers. The single-particle-pair correlation function has been evaluated and shows significant structural features up to 15 Å. It has been tentatively assigned upon results from Monte-Carlo simulation. The density decrease at high temperatures as well as the displacement of the conformational equilibrium are detected and analyzed.

## Dynamics of amorphous solids

Time-of-flight (TOF) measurements on amorphous $Ge_xSe_{1-x}$ have been carried out on IN6 (Elliot, Gladden, Wright, Sinclair, Dianoux) and on IN4 (Kamitakahara, Gompf, Cappelletti, Mutka) to study the vibrational density of states. The IN6 experiments give evidence for a low frequency intensity enhancement with decreasing Ge concentration. The Q-dependence of the inelastic intensity of $GeSe_2$ indicates coupled tetrahedra rotations similar to quartz (Buchenau et al.). The IN4 results show the drastic changes in the vibrational density of states for pure Se and for increasing Ge content ($x < 0.33$). Special low frequency "floppy" modes are predicted by the theory (Thorpe et al.), which should disappear for samples with coordination numbers greater then 2.4. The observed smooth variation of the low energy intensity with concentration seems to be in contradiction to the prediction of the onset of rigidity percolation.

## Glass transition

The study of the glass transition has been a main topic of the ILL Workshop on Dynamics in Disordered Materials. The neutron

experiments in this field had been mainly continuation work on different materials or on different instruments in order to get a wide experimental dynamical range. Furthermore, the investigation of a wide temperature range is essential.

Of main interest is a comparison of the experimental results with the mode coupling theories. Even, if there is some experimental confirmation of several of the theoretical predictions, the experiments up to now neither clearly confirm nor disproof the validity of the mode coupling theory near the glass transition. In the following only one example, out of many studies done in the last year, is given (Richter, Frick, Farago).

As a consequence of structural irregularities very often polymers are good glassformers and do not crystallize even under slow cooling. Simple polymers, therefore, are well suited to study the dynamics around the glass transition. The microscopic dynamics of deuterated trans-cis-vinyl (47:46:7) polybutadiene, a polymer consisting only of a backbone without sidegroups, was investigated on the scale of typical interchain distances by measuring the time dependent intermediate scattering function in the neighbourhood of the interchain peak in the structure factor S(Q).

Fig. 60 presents the results obtained at the peak position of S(Q). According to the time temperature superposition principle the data are presented in a scaling representation. Thereby the scale $\tau$ has been obtained from viscosity measurements. For more than 7 orders of magnitude in the rescaled time $t/\tau$ the spin-echo data follow a Kohlrausch law with a time exponent $\beta = 0.45$ following a temperature scale derived from **macroscopic viscosity** measurements. Fig. 60 shows that for $t/\tau \rightarrow 0$ S(Q,t)/S(Q,0) does not extrapolate to one but approaches a value distinctly smaller than 1 ($0.917 \pm 0.008$). This observation shows that at the glass transition only a fraction of the dynamic structure factor becomes non ergodic, while a substantial part does not freeze. Finally, the insert in Fig. 60 presents the temperature dependence of the non-ergodic fraction measured at the peak position of S(Q). Above 200 K f(Q) measures the spectral contribution of the structural relaxation. Its value fluctuates

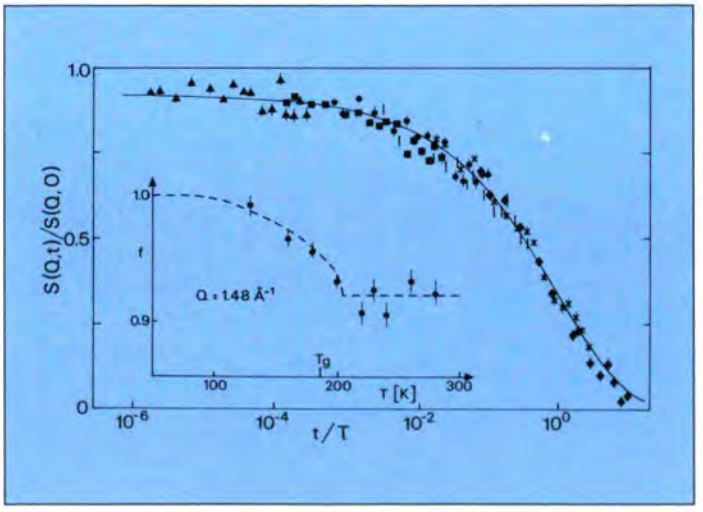



*Figure 60* *Scaling representation of the spin echo data for $Q = 1.48$ Å$^{-1}$ (▲: 200 K, ■: 220 K, ●: 230 K, ❙: 250 K, ✖: 260 K, ◆: 280 K). The scale $\tau(T)$ is taken from a macroscopic viscosity measurement. Insert. Temperature dependence of the non ergodicity parameter f(Q) near the maximum of S(Q) ($Q = 1.48$ Å$^{-1}$).*

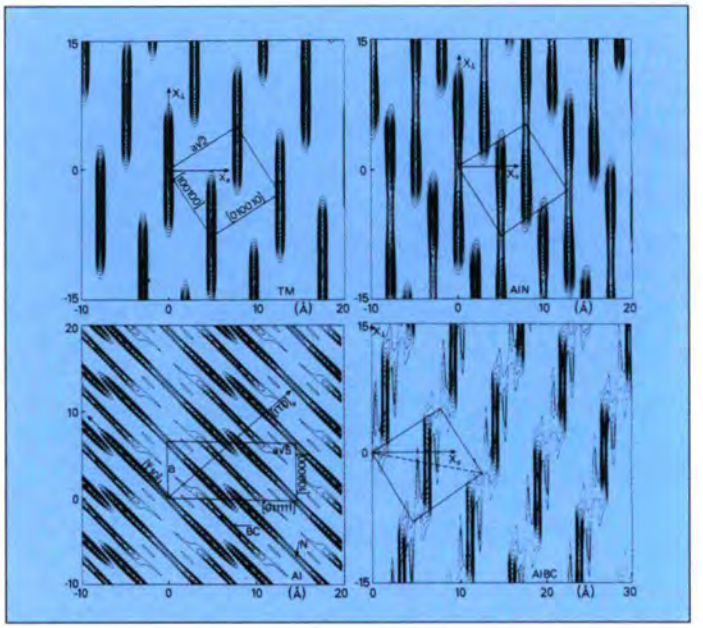



*Figure 61* *Samples of 2D-cut of the 6D periodic partial atomic densities. The transition metal site is unique. At the nodes of the 6D cubic lattice its acceptance function "decorates" the site. There are two different aluminium sites, one at the nodes and the second one at the body centres, also "decorated" with the pertinent acceptance functions. The cut sections of the 6D cubic unit cell are shown along with the co-ordinate axis of the cut. TM stands for the transition metal atom sites, AlN and AlBC for aluminium node sites and body centre sites, respectively. The AlBC node shown here is the (3/2,1/2,1/2,1/2,1/2,1/2) one corresponding to $x_{//} = 6.33$, $Y_{//} = 7.82$, $Z_{//} = 3.92$, $X_{\perp} = -1.49$, $Y_{\perp} = 4.82$ and $Z_{\perp} = 2.42$ (in Å).*

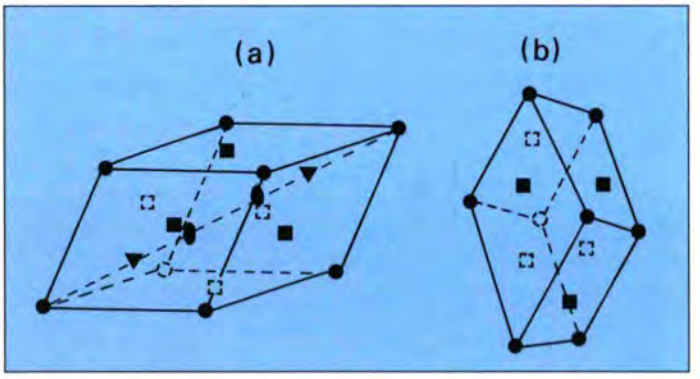



*Figure 62* *Schematic presentation of the atomic decoration of the prolate (a) and oblate (b) rhombohedra.*

●, ○ *Transition metal site corresponding to node sites in 6D with a pseudo-spherical acceptance function,*

□, ■ *Al sites on the long diagonals of the faces,*

▼ *Al sites on the triad axis*

□, ■, ▼ *Correspond to node sites in 6D with a pseudo spherical shell as an acceptance function),*

⬮ *Al sites on the triad axis corresponding to the body centres in 6D with a very diffuse spherical distribution as an acceptance function.*

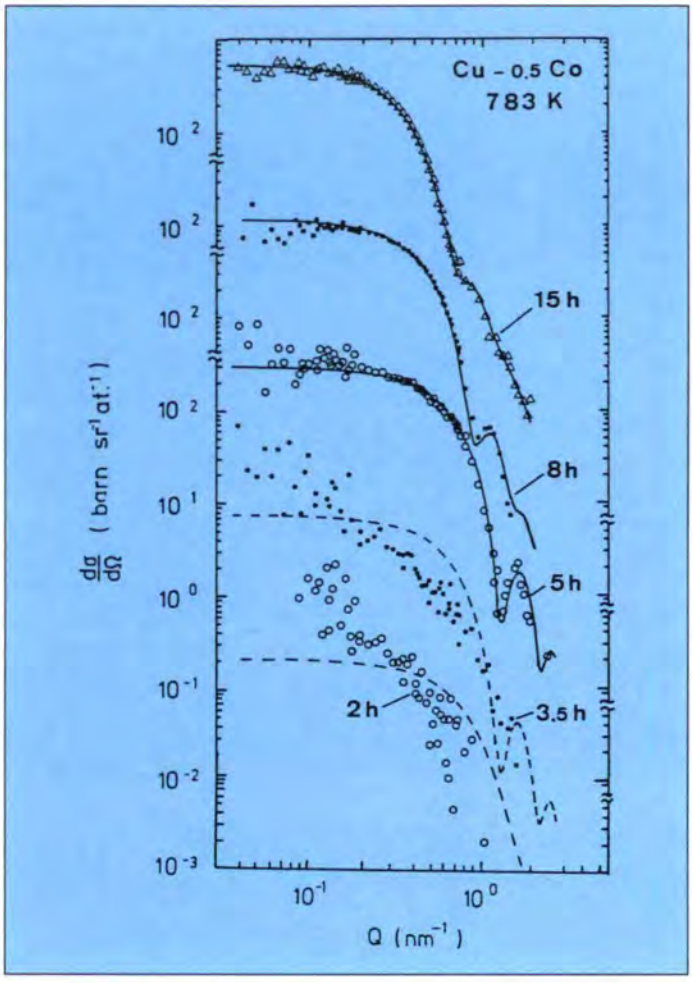



*Figure 63 SANS structure functions of Cu(0.5 at.% Co) after annealing at 783 K. The structure function of the reference sample (2h at 1223 K; water quenched) has been subtracted. Solid and dashed lines: calculated structure functions of spherical α-precipitates, fitting well (solid line) or unsatisfactorily (dashed line) to the data.*

around the result from the $t/\tau \rightarrow 0$ extrapolation of the data and basically stays constant. Below about 200 K the structural relaxation freezes and becomes non ergodic. Here f(Q) measures the non ergodicity. We note that f(Q) increases gradually and reaches its limiting value 1 only far below the thermodynamic glass transition ($T_g$ = 85 K). Thus fast motional processes at a time scale above $10^{-10}$ s but below $10^{-13}$ s become effective already well below $T_g$.

## Quasicrystals

After about two years of measurements at the ILL and deep thinking on the best way to use diffraction data in the case of non-periodic structures, the first experimentally determined structure of quasicrystals (QC) has been obtained by combining high resolution powder measurements (D2B) and investigations up to very large Q values (D4).

Using well tested isomorphous substitutions on the manganese atom sites of an Al-Mn-QC to produce a contrast variation effect, it has been possible to measure amplitudes and phase differences of the partial structure factors for the individual Al and Mn subnetworks. The data were checked to be consistent with a hidden periodicity in a 6-dimensional space which enabled a successful phase reconstruction procedure to be elaborated and the subsequent calculation of the partial atomic densities either in our 3D physical space or the 6D related periodic structure to be completed (Fig. 61).

This structure in 3D corresponds to space filling with two different rhombohedral cells (prolate and oblate) assembled to form a so-called 3-dim Penrose tiling (3DPT) with edge length equal to about 4.6 Å. The atomic decoration of this 3DPT can be described as follows (Fig. 62):

1) The Mn atoms are sited at vertices only.

2) The Al atoms are distributed in space with more disorder, mostly at points dividing the long diagonals of faces into segments of about 3 and 4.8 Å, and also in two types of sites on the triad axis of the prolate rhombohedra at 2.6 and 6.8 Å of the vertices. In a given rhombohedron all these sites are never occupied simultaneously and actual positions are

somewhat scattered around the average theoretical sites. Matching rules for the oblate and prolate rhombohedra are related to occupancy of the face sites.

Such a description is in fact not surprising at all. A rigid non periodic tiling model, with all the tiles decorated with strictly equivalent sites actually cannot be accepted. Such a scheme would result in unstable structures due to the existence of incompatible bond angles and pair distances. The transition metal substructure propagates the long distance ordering of the whole structure while the aluminium atoms introduces the necessary "chemical modulation" permitting stability.

## Metal physics

Small-angle neutron scattering experiments on CuCo alloys containing 0.5 and 0.8 at.% Co gave evidence for precursor stages of decomposition prior to the α-phase precipitation (Wagner, Petry) (Fig. 63). Compositional fluctuations which are large in extent and small in amplitude compared to the subsequently forming α-precipitates were observed. In the presence of such compositional fluctuations the conditions for application of mean field theories for nucleation should be disturbed. In fact the SANS data reveal nucleation rates significantly higher than theoretically predicted, e.g. a factor of four higher for the 0.5 at.% Co alloy and the smallest stable α-precipitates being up to 5 times as large as the theoretically expected size of critical nuclei. The latter gives rise to an unusally sharp precipitate size distribution. This is reflected in the second order maximum resolved after annealing.

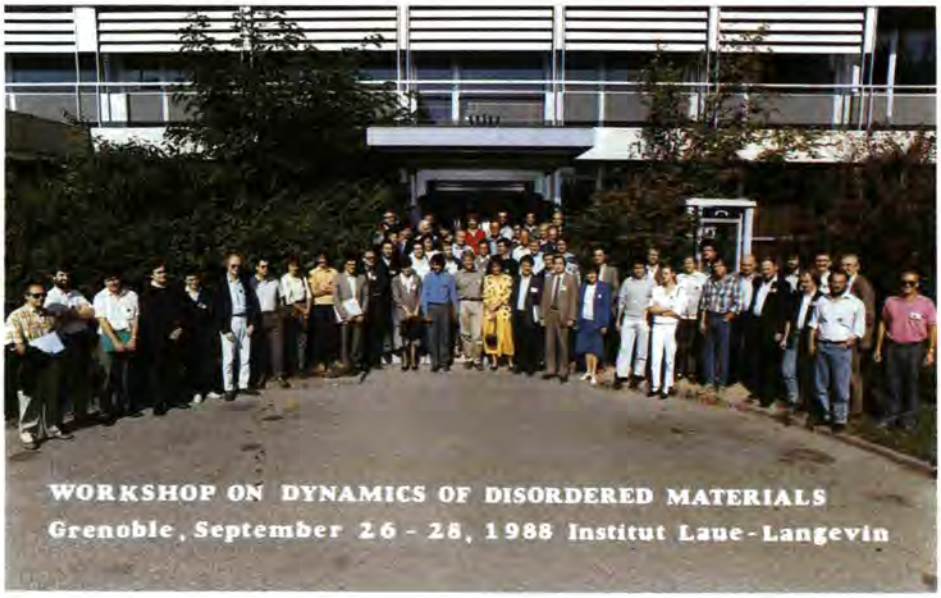
## Hydrogen in metals

The study of **hydrogen tunnelling** requires a rather dilute system (less than ca. 0.2 at.%) with impurity atoms present to prevent hydrogen precipitation when the sample is cooled. Despite these difficulties, it is possible to compare the tunneling spectra of $Nb(OH)_x$ an $Nb(CH)_x$. Data for the oxygen analogue have been available for some time, but more recently, data for $Nb(CH)_x$, with x at only 0.0002, have become available (Fig. 64). The tunnel splitting of the $Nb(CH)_x$ is found to be considerably lower than that of $Nb(OH)_x$. This is an indication that the impurity atom influences the potential, which is of importance to the general understanding of hydrogen diffusion in these systems (Wipf, Steinbinder, Kearley).

According to Kondo, at low enough temperatures, the **quantum diffusion** of light interstitials in metals is controlled by direct, nonadiabatic interaction with conduction electrons. This interaction results in jump rates for the interstitials that exhibit a $T^{2K-1}$ temperature power law, where K is a dimensionless coupling parameter. A study of the local jump diffusion of trapped H in $Nb(OH)_x$ samples between 10 K and 160 K on IN6 could show that the jump rates vary less than a factor of seven and that they exhibit below $T \cong 70$ K Kondo's power law. Above this temperature the diffusion is mainly dominated by the interaction with phonons (Fig. 65). (Steinbinder, Wipf, Magerl, Richter, Dianoux, Neumaier).

**High temperature diffusion of hydrogen** in $YH_x$ has been studied on IN10 between T = 450°C and 950°C and for H concentrations between x = 1.8 and 1.97 (Stuhr, Schlereth, Steinbinder, Wipf, Frick, Magerl) and on $V_{0.25}$ $Nb_{0.75}$ $H_{0.2}$ and $YH_{1.98}$ (Barnfather, Styles, Dianoux). The results from both groups are in contradiction to recent NMR results where a decreasing diffusion constant with increasing temperature had been reported. The IN10 results show an activated behaviour with increasing temperature and an increase of the diffusion constant with increasing concentration. This may be explained by a partial occupation of the octahedra sites by hydrogen atoms.

A **new class of hydrides,** synthesized under high H-pressure (up to 90 kbar), with subsequent quenching, has been measured on the IN1-Beryllium filter instrument. During a long time palladium hydride was the only representative of hydrides from transition metals belonging to the VI-VIII group of the periodic system. The reason was that only this hydride could be produced under equilibrium conditions. The new class contains hydrides of Ni, Cr, Mo, $V_{0.9}$ $Ru_{0.1}$, $Nb_{0.75}$ $Ru_{0.25}$. Fig. 66 shows an example from the first results on IN1. (Antonov, Belash, Bokhenkov, Poniatovsky, Pronina, Dorner).




*Two very exciting Workshops on Quasicrystals (left), and on the Dynamics of Disordered Materials (right) were held at the ILL from 21 to 25 March and from 26 to 28 September 1988 respectively. Many participants attended lectures on these very timely topics.*

## Ionic solids

The **solid electrolyte** actually used in the electrochemical Na/S batteries is mainly the β" alumina. This compound is a rhombohedral variety of the β alumina compound; its ionic conductivity, depending on the sample treatment, can be ten times higher than the β variety. Quasi-elastic neutron scattering experiments (IN6) on a single crystal of $Na\beta"Al_2O_3$ to investigate the mobility (Fanjat, Lucazeau, Bates, Dianoux) of $Na^+$ gave:

i) a larger quasi-elastic intensity in β"-than in β-alumina, indicative for either larger jump distances or a larger number of mobile $Na^+$ ions in β"- than in β-alumina;

ii) no significant variation of the width of the quasi-elastic scattering with Q, indicative for local motions (in both β- and β"-alumina);

iii) a narrower width of the quasi-elastic line, i.e. slower motion, in β" than in β-alumina. These results can be understood from models for the two dimensional ionic conductivity.

Secretary : B. Frick

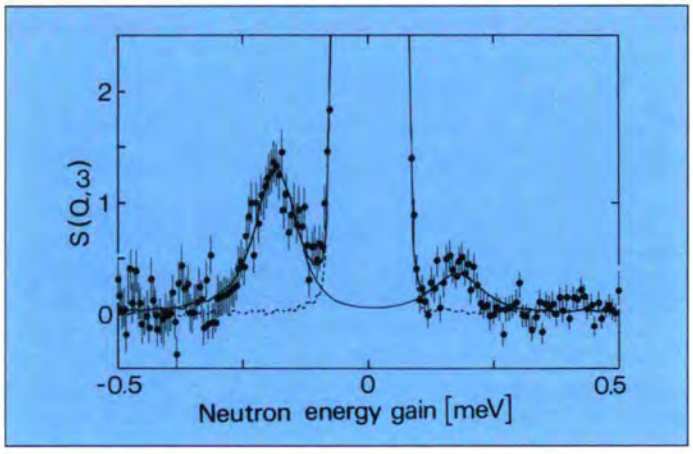



*Figure 64 Hydrogen tunneling spectrum of $Nb(CH)_x$, where $x = 0.0002$ measured on IN5 at $T = 1.8$ K.*

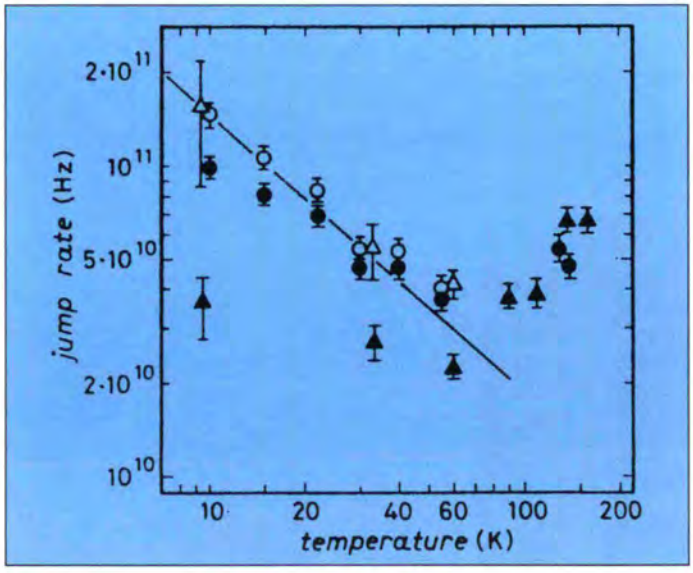



*Figure 65 Jump rates of H in two $Nb(OH)_x$ samples. The circles are the data points for $x = 0.002$ and triangles for $x = 0.011$. The full line gives the theoretical prediction according to Kondo's power law.*

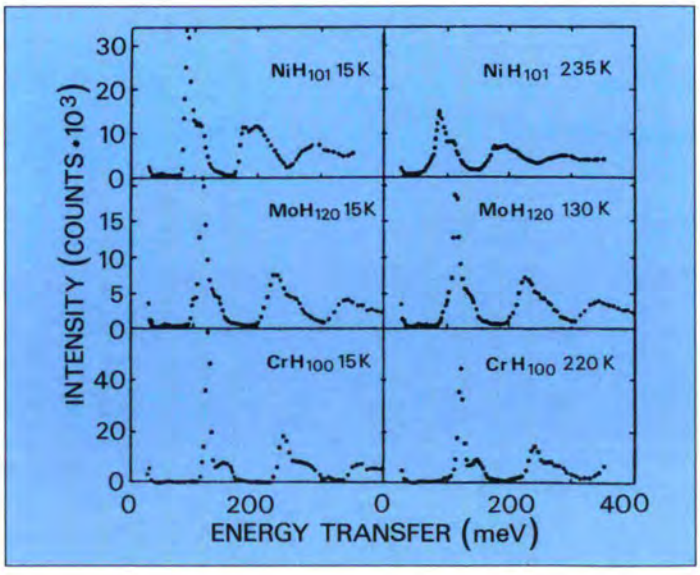



*Figure 66 Example of vibrational spectra for a new class of hydrides measured on the IN1-Beryllium filter instrument.*

# Small-Angle Scattering from Fractal Systems

C. Janot

## Introduction

Compared to classical Bragg diffraction, neutron scattering at small angles, or rather at small scattering vectors Q, is mainly related to microstructural entities instead of individual atoms. Consequently, the technique is currently used to investigate size and distribution functions of aggregates, voids, small precipitates, magnetic heterogeneities, etc.

The small-angle scattering from many heterogeneous disordered or porous systems is found to include so-called "power laws" in Q and this suggests that the data be interpreted by assuming that it arises from a scale invariant (or fractal) structure. The fractal nature of such systems is a statistical property which needs to be averaged over macroscopic sample volumes. Thus, in this prospect, it is very fortunate that in small-angle neutron scattering (SANS), the high penetrability of the neutrons allows statistical properties representative of the whole sample to be obtained.

## Notion of fractal geometry and scaling

As a simple **non-random** fractal model, let us consider the example of the so-called Sierpinski gaskets. Fig. 67 shows four stages of the recursive construction of the two-dimensional case. At each iteration, the length scale is divided by 2 and each triangle is replaced by three smaller black ones surrounding a cut out central small (white) triangle. As the recursive construction is repeated (in principle *ad infinitum)* the resulting structure is a good simulation of a perfectly ordered fractal or self-similar system.

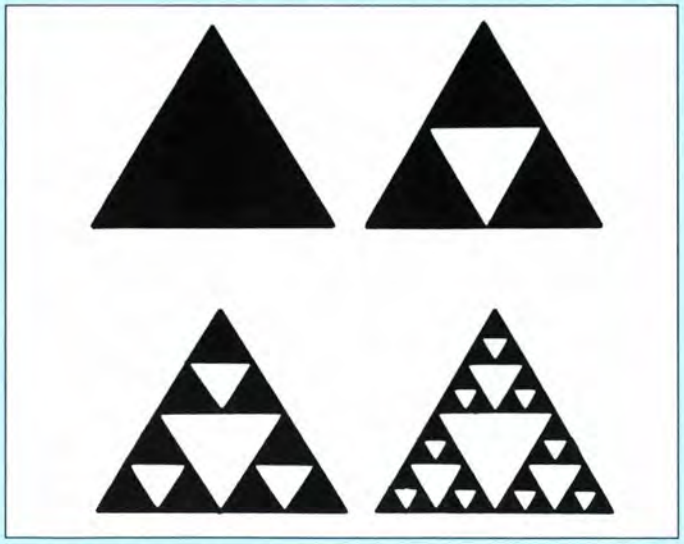


*Figure 67 Four stages of iteration of the so-called Sierpinski gasket as an illustration of ordered fractal structure. The iteration procedure can be repeated further.*

The behaviour of such a fractal structure has to be contrasted with that of a homegeneous system in which the mass (or filled volume) scales within a box of linear size L as a power law $M(L) = \rho L^d$, where d is the Euclidean dimensionality (2 in Fig. 67) and $\rho$ uniform density. In the last stage of the recursive construction shown in Fig. 67, the largest triangle has a filled (black) surface which is three times that of the preceding intermediate triangle, which in turn is three times as "heavy" as its own preceding intermediate one, etc. Thus, the mass (or filled volume) is multiplied by 3 (instead of 4 for homogeneous system) when the linear size is multiplied by 2. Then it scales as

$$M(L) = \bar{\rho} L^D \qquad (1)$$

with $2^D = 3$ or $D = ln\,3/ln\,2 = 1.5849...$

For a d-dimensional generalization, using tetrahedra or hyper-tetrahedra instead of triangles, it is straight forward to demonstrate that, for $d < 6$:

$$D = ln\,(d+1)/ln\,2 \qquad (2)$$

D is called fractal dimensionality.

From the simple case of Fig. 67, it is also clear that a system may appear as fractal or not, depending on the observation scale with respect to the length scale of the system. A panoramic view of a many-iterated Sierpinski-gasket looks indeed fractal but a short distance observation focused on one of the smallest black triangle revealed a mere homogeneous system. Similarly, if observed from far too large a distance, the system also appears as a unifom grey patch. Consequently, scale invariance as expressed by Eq(1) may be expected to break down, with a cross-over from fractal to non fractal behaviour, at both "small" and "large" sampling of the system.

Finally, it can be demonstrated that the notion of fractal dimensionality readily extends to disordered heterogeneous self-similar systems with, for instance, exponent D very close to 1.9 at $d = 2$, to 2.5 at $d = 3$ and equal to 4 at $d > 6$ when percolation models are used.

## The SANS approach to three-dimensional fractal systems

Let us consider, for the sake of simplicity, an heterogeneous system which may be described in terms of a two-phase model. The scattering entities causing SANS are particles with similar shape, size and chemical composition, embedded in a matrix. The differential macroscopic SANS cross-section $d\sigma/d\Omega$ for such a two-phase system is given by interferences of the beam scattered by each particle and thus may be expressed as:

$$\frac{d\sigma}{d\Omega} = f\left(\rho_p - \rho_m\right)^2 S(Q)\, F^2(Q) \qquad (3)$$

with f = volume fraction of the scattering entities, $\rho_p$ = coherent neutron scattering length density of these scattering entities, and $\rho_m$ = mean coherent neutron scattering length density of the background matrix. $F^2(Q)$ is

the one-particle term and is related to the shape (form factor) of the scattering entities. S(Q) is an interference function related to the pair correlation function of the particles.

Actually, Eq(3) applies to any two-phase system, including fractal structures. In that case, $F^2(Q)$ is simply the form factor of the smallest building blocks in the system (for instance, the smallest black triangles shown in Fig. 67). If the observation scale is to match the scale invariant size range or, in other words, if the investigated Q range corresponds to the observation of a "large" sample of the fractal structure, at least large enough to validate the definition of an average pair correlation function, then each of these smallest building blocks may be considered as almost point features and their form factor $F^2(Q)$ does not vary significantly over the Q range of interest. Within assumptions of ergodicity (memory effects over time dependence), homogeneity (space equivalence of particle positions) and isotropy of particle distributions, the interference function S(Q) is given by:

$$S(Q) = n\int_0^{\infty} 4\pi r^2 g(r) \frac{\sin Qr}{Qr} dr \qquad (4)$$

where g(r) is the pair correlation function for the smallest building blocks arranged into the volume fractal structure and n is the average particle density. If Eq(1) is readily used to express the member of entities $N_v(R)$ within a given spherical box of radius R, namely $N_v(R) \propto R^{Dv}$ with $D_v$ the volume fractal dimensionality, g(r) has then to fulfill the relation:

$$\int_0^{R} 4\pi r^2 g(r)\, dr \cong R^{D_V} \qquad (5)$$

and hence: $g(r) \propto r^{Dv-3}$

In the above derivation, only the general trend of $N_V(R)$ and g(r) are considered. These functions may obviously have oscillatory r dependences which are not accounted for here and, actually, have no influence on the forthcoming conclusions. The g(r) function as expressed by Eq(5) is of course valid only for the r range where the scale invariance exists. At large $r \geq \xi_v$ (maximum volume correlation length) the scale invariance breaks down and the system appears as an homogeneous one. This can be allowed for by writing:

$$g(r) \propto r^{Dv-3} \exp(-r/\xi_V)$$

At small r the scale invariance must also break down as two elements cannot approach to within a centre to centre distance of less than $2R_g$ (twice the radius of gyration of the smallest building blocks). Then $g(r) = 0$ for $r \leq 2\ R_g$.

Finally, using Eq(3) (4) (5) it is trivially easy to demonstrate that the SANS cross-section $d\sigma/d\Omega$ should have a $Q^{-D_v}$ behaviour in the range $2QR_g \leq 2\pi \leq Q\xi_v$. Then, plots of *ln* $(d\sigma/d\Omega)$ vs *ln* Q derived from SANS data may give $D_V$, $R_g$ and $\xi_v$. As an example of such a derivation, Fig. 68 shows the *ln/ln* plot of small angle scattered intensities for a wet titania gel (from Marignan, Guizard and Larbot, to be published in Europhysics Letters). The classical $Q^{-4}$ Porod behaviour is preceded by a segment of slope -2.08 extending roughly from $Q = 10^{-2}$ to $10^{-1}$ Å$^{-1}$ i.e. $R_g \cong 30$ Å and $\xi_V \cong 600$ Å.

There is another kind of scale invariance which needs to be considered in a three-dimensional system, namely a so-called "surface fractal" structure in which, for three-dimensional features, the surface has a fractal dimensionality $D_S$. For a smooth ideally perfect surface $D_S = 2$. For a real surface a certain chemical or/and topological roughness must be considered due to composition profiles, porous layers, voids and scratches, etc. Then either the free surfaces or the interfaces between the two partners of the two-phase system may actually have a "thickness" and behave as a 3D medium with $D_S = 3$. Qualitatively it can be also understood that surface roughness may exhibit self similar heterogeneous disordered structure with intermediate (fractal) dimensionalities $2 \leq D_S \leq 3$. Such "rough" surfaces have also a measurable SANS cross-section. In the particular case of a distribution of spherical scattering particles of various sizes in a matrix, with perfectly smooth surfaces ($D_S = 2$), the classical Porod law already mentioned expresses that the SANS cross-section drop off as $Q^{-4}$, i.e.:

$$\lim_{Q\to\infty} \frac{d\sigma}{d\Omega} \approx \frac{2\pi(\Delta\rho)^2}{Q^4} \cdot \frac{S}{V} \qquad (6)$$

Here S is the total surfaces area of the spherical scatterers in the volume V of the sample exposed to the beam; $\Delta\rho = \rho_\rho - \rho_m$. Eq(6), when easily generalized to cases of fractal interfaces, shows that the SANS signal then behaves as $Q^{Ds-6}$ over the Q range corresponding to the scale invariance alike for a volume fractal structure. Conclusively, surface fractal structures are expected to produce *ln/ln* plots with slopes ranging from -4 to -3. It is worth mentioning that this analysis applies as well to both intersurfaces (internal surface separating two phases within the sample) and external surfaces.

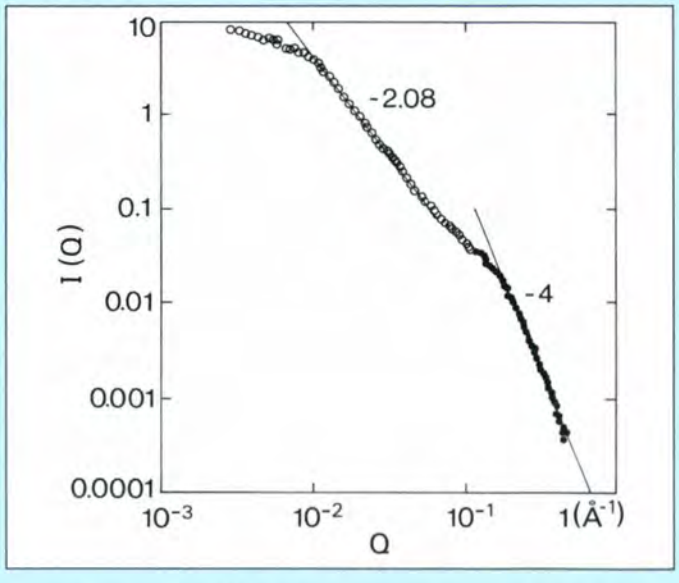



*Figure 68 Small angle scattering intensity measured with a wet titania gel. A clear cross-over from fractal to non fractal behaviour shows up at scattering vector about 0.1 Å$^{-1}$.*

As an example to illustrate the point, Fig. 69 reproduces a *ln/ln* plot of the SANS intensity coming from the free surfaces of ribbons of a melt quenched iron-based amorphous alloy.

SANS experiments have for several years been expected to produce the information needed concerning medium-range order, density and composition fluctuation in amorphous alloys. Detailed interpretation in term of defects, namely quasi-dislocation effects, has even been proposed to account for the dependence of the scattered intensity on a negative Q power law. This might have been jumping too fast to a desired conclusion. Obviously, amorphous ribbons with about 30 µm thickness have quite a large surface-to-bulk ratio and surface imperfections have been known to contribute to SANS signals. Additional scattering may come from magnetic heterogeneity if iron-based glasses are concerned. Thus, investigating bulk heterogeneity in amorphous alloys using the SANS approach is not really straightforward and careful analyses of both magnetic and surface effects have to be carried out before concluding to the existence of quasi-dislocations or other bulk influences.

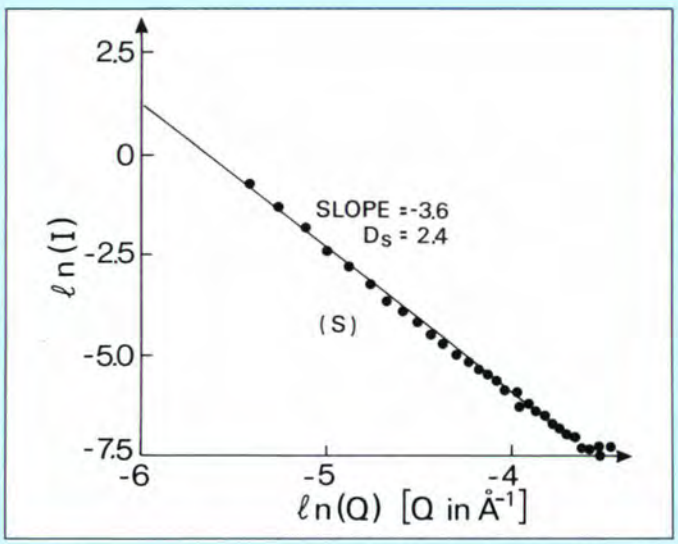



*Figure 69 Double logarithm plot showing the surface fractal structure for ribbons of iron-based amorphous alloys.*

The SANS scattering part due to possible magnetic heterogeneity can be eliminated by applying a strong magnetic field on the sample (saturation effects). An elegant method to get rid of the scattering due to surfaces states or imperfections is to immerse the sample into a liquid whose scattering length density matches that of the sample, so that any contrast between the sample and the outside is annihilated. For neutrons it is as if voids, scratches, etc. on the surface were filled with the same material as the sample itself.

As an example, amorphous $Fe_{70}Cr_5P_{15}C_{10}$ amorphous alloys have been investigated using SANS. In this particular case, the average scattering density of the sample matches very well the one of heavy water $D_2O$. SANS signal from the sample in zero magnetic field and without heavy water in the containing cell includes possible contributions from the bulk, the surface and magnetic inhomogeneity. In the presence of a sufficiently large magnetic field, but still without heavy water in the cell, we are left with atomic inhomogeneity and surface imperfections. SANS with the applied field and with the heavy water gives the pure contributions from atomic bulk inhomogeneity (both density and concentration fluctuation). In conclusion, it is possible to extract the three individually mentioned contributions to the SANS signals from a set of three experiments on each sample. This is precisely how the data shown in Fig. 69 have been obtained. In this particular case, the *ln*(I) vs *ln*(Q) plots fit very well to a straight line with a slope - 3.6 which suggest a fractal structure of the ribbon free surfaces. The deduced fractal dimensionality would thus be $D_S = 2.4$. Incidently, it is also worth mentioning that, contrary to what has been claimed very often, the SANS fraction coming from bulk non-magnetic heterogeneities is very small. The main part (95%) comes from magnetic effect and about 5% is due to free surfaces. Less than 0.1% can be attributed to bulk atomic heterogeneities.

## Conclusion

In the present contribution, we have tried to illustrate the suitability of SANS measurements to approach microstructural problems related to fractal dimensionalities. More quantitative models can even be tailored to lead to realistic parameters when it is not possible to apply a discrete particle size distribution description of the microstructure. Both volumes and surfaces may have fractal dimensionalities and, practically, it is not always easy to separate the corresponding scale invariance which contributes to the SANS signal. However, imperfect as it is, the obtained information is unique most of the time.

# Biological Structures and Dynamics

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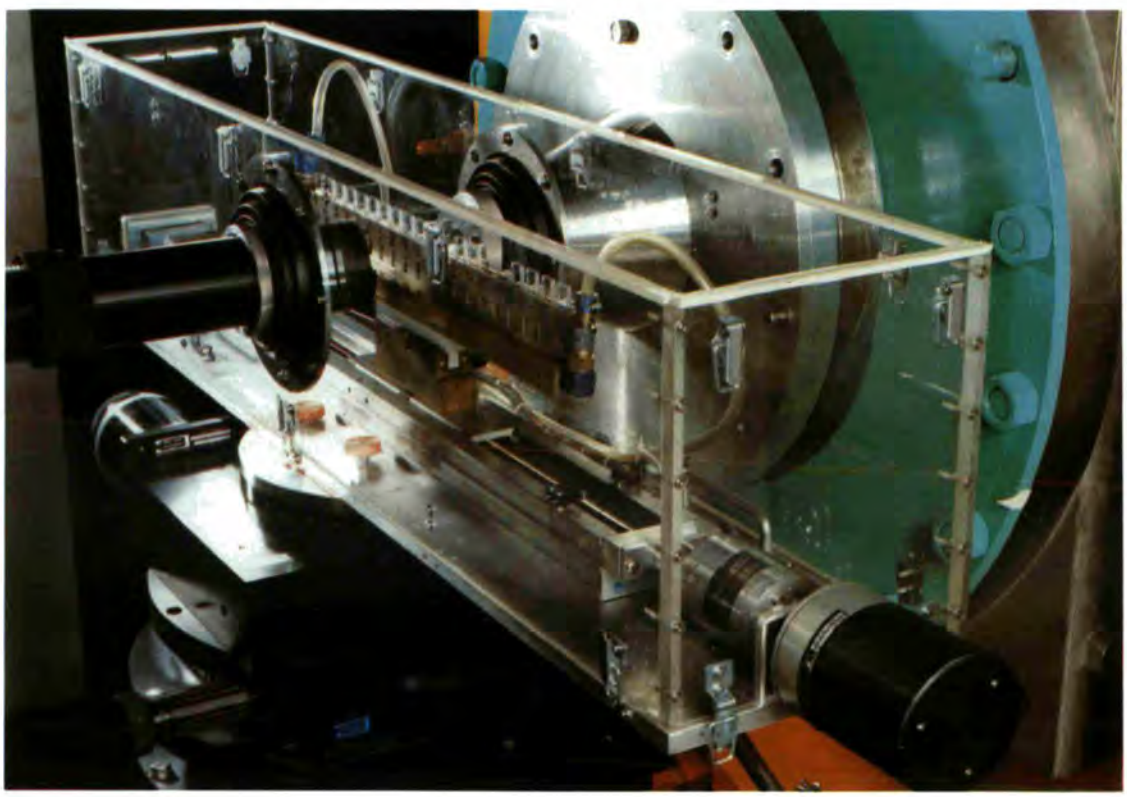
## Introduction

Neutron scattering is an unique tool for studying certain aspects of biological structures and dynamics. The largest number of proposals in College 8 is for the use of the small-angle scattering instruments D11 and D17 to study the structures of biomolecular complexes in solution using the contrast variation technique. In addition a number of experiments on partially ordered systems such as membranes and single crystals, both at high and low resolution, have been carried out on D16 and DB21. Inelastic scattering, which in other scientific areas is a well established technique, is beginning now to be developed into a useful tool for understanding the dynamics of biological macromolecular systems. The following section describes some of the projects carried out during 1988 but is by no means exhaustive.

## Scientific Trends and Highlights in 1988

### Protein-nucleic acid interactions

Protein-nucleic acid interactions are central to a vast range of cellular processes including gene regulation, recombination, protein synthesis and assembly/disassembly processes. Due to the large difference in scattering density between proteins and nucleic acids the contrast variation technique provides a powerful method for the investigation of such structures both in solution and the crystalline state.



*The sample changer of the D11 small angle scattering facility operates in the temperature range –5 to 95°C and can accept 20 samples at any one time.*

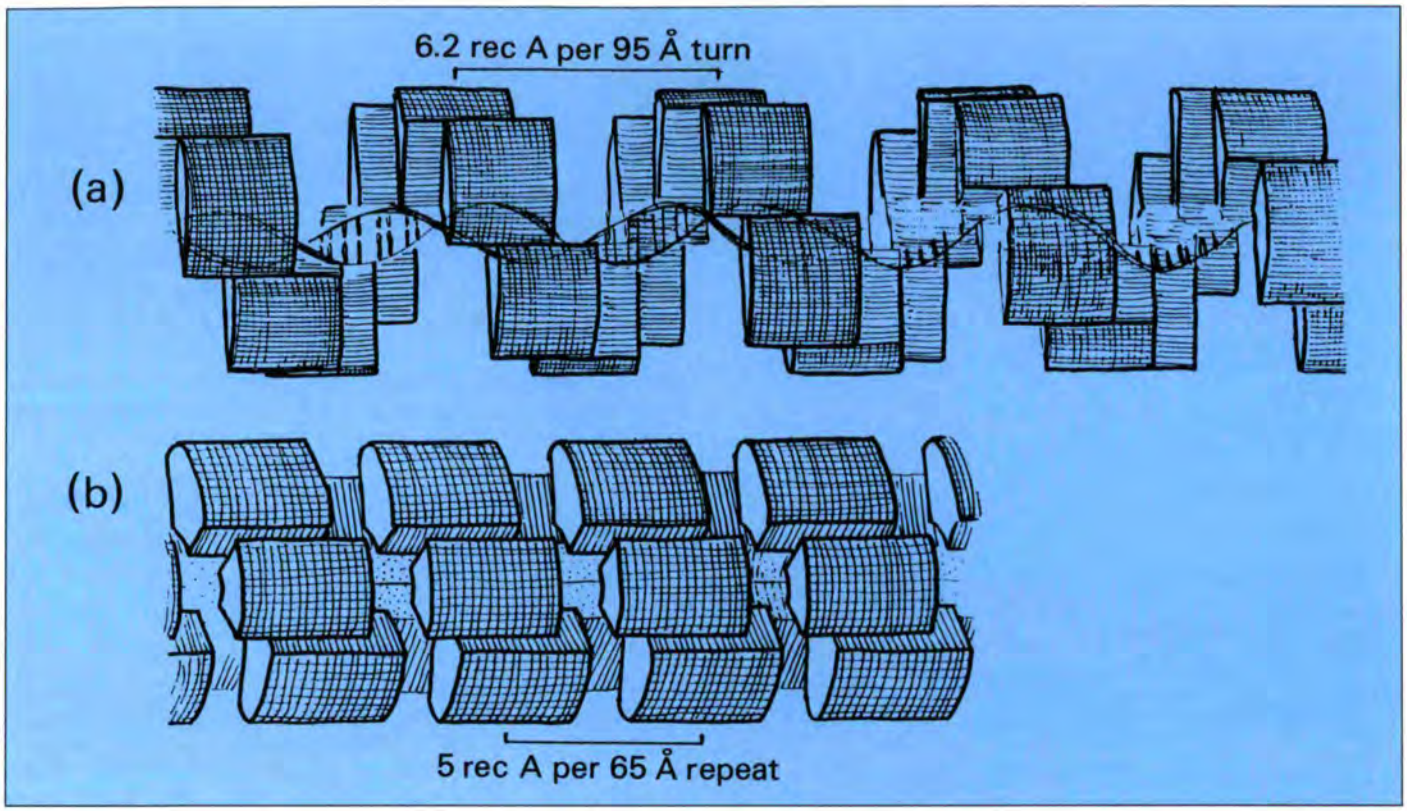



*Figure 70 Models for two different forms of the recA polymer. (a) Extended form as observed in complexes with DNA in the presence of ATPγS. (b) Compact form as observed for the recA self-polymer.*

The Tet repressor controls transcription of the tet genes conferring resistance to tetracycline, an antibiotic with importance for human therapeutic use. The knowledge of the spatial arrangement of the Tet repressors on the tet gene control region is important to understand the regulation of the expression of the proteins responsible for the resistance to tetracycline. In the repressed state, two Tet operators are occupied by one Tet repressor dimer each. The spatial arrangement of the two Tet repressor dimers on the wild type Tet gene control region has been determined recently by neutron small-angle scattering (Max-Planck-Institut Martinsried, ILL). The centre-to-centre distance between the repressor dimers was measured as 110 ±5 Å. The distance distribution function shows that they have no direct contact, but maintain a minimal distance of 50 ±20 Å. Contrary to other regulatory systems, control in the Tet system can therefore not be mediated via direct protein-protein interaction.

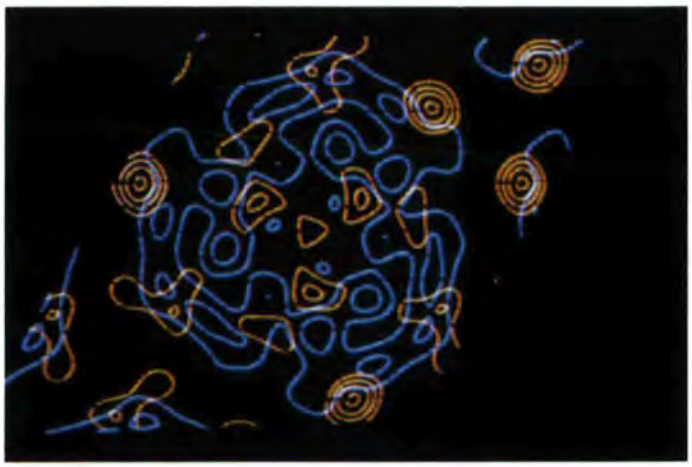
The recA protein from *E. Coli* is able, *in vitro*, to promote strand exchange between homologous DNA molecules. The purified protein is a rod shaped polymer which, upon binding to single stranded DNA, displays two different appearances depending on the presence or absence of ATP (or its non-hydolyzable analogue, ATPγS). Binding to double stranded DNA occurs only in the presence of ATP. Neutron small-angle scattering has been used to complement the findings of electron microscopy and biochemistry (ILL, EMBL, IBMC Strasbourg), indicating a mass per unit length compatible with 6.2 recA / 100 Å for the complex with either type of DNA in the presence of ATPγS. The neutron results highlight the difference between two types of recA polymers; on the one hand the ATP induced structure of the complex with double- or single-stranded DNA and on the other the homopolymer and the single-strand complex in the absence of ATP (Fig. 70). They differ by 20% in mass per unit length as well as in their cross-sectional radii of gyration (32 Å vs. 40 Å). The binding of ATPγS alone to the polymer was shown not to elicit these changes, providing evidence that the strong conformational change in recA is due to the synergistic effect of both ligands, DNA and ATP.

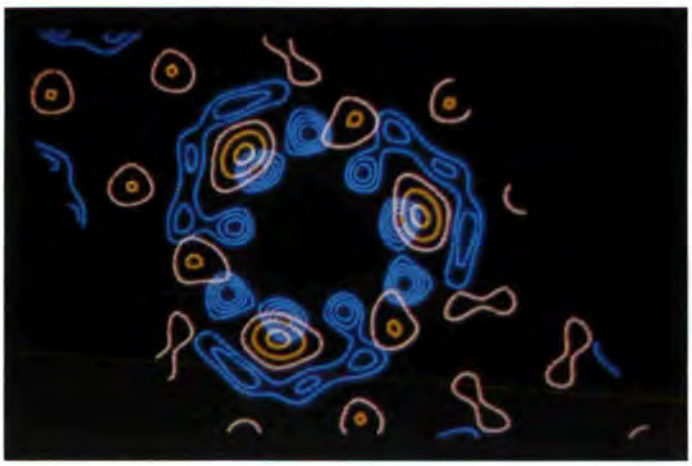
DNA gyrase catalyzes the ATP-dependent supercoiling of DNA. Neutron scattering data were combined with information from complementary techniques to construct models of bacterial DNA gyrase and its complex with DNA (Leicester, NBS and NIH (USA), ILL).

Ribosomes are the protein synthesizing machinery in all living cells. They are large molecules (molecular mass ≈ 3 million) consisting of two subunits of different size in all organisms. Both of them are composed of ribonucleic acid (RNA), making up 2/3 of the ribosomal mass, and a multitude of proteins. The large ribosomal subunit from *E. Coli*, which can easily be grown in many different culture media, consists of two RNA chains, a very long and a short one, and more than 30 different proteins.

A long-term small-angle scattering study is being conducted by scientists of the Max-Planck-Institut für Molekulare Genetik, Berlin, and the ILL to investigate the spatial arrangement of the ribosomal proteins within the large subunit. Following a

positive review by external experts in early 1988, the ILL is continuing its committment to this project. Meanwhile more than 100 data sets for inter-protein scattering curves collected over the years have been retreated with a new program fitting the corrected pair scattering curves by the Fourier transform of a pair distance distribution function which is defined by a set of spline functions (smooth functions composed of polynomials of degree 3 for approximating peaks) with variable position, width and height in real space. The distance information obtained by this procedure yields the mass-centre positions of 16 proteins within the large ribosomal subunit, and it will soon be possible to add about 6 more proteins to this map.





*Figures 71 & 72*
*Purple membrane neutron difference maps. The red contours represent in 71 the positive difference between a reconstituted sample with deuterated leucines and the native and in 72 the positive difference density between the native and the C-terminus cleaved structures. Superimposed are blue contours representing the projection of the native bacteriorhodopsin structure.*

Experiments have been continued on DB21 on single crystals of 50S ribosomal subunits from *Halobacterium Maris Mortui* (Berlin, Hamburg, LURE, EMBL, ILL). This work has been facilitated by a new computer program (written by P. Metcalf, EMBL) which allows very easy examination of the diffraction data on a PS300 Evans & Sutherland graphics terminal. Through a simultaneous representation on the screen in reciprocal space of the measured reflections and the reciprocal lattice of the crystal the orientation matrix of the crystal can be rapidly and efficiently determined.

Seryl-tRNA synthetase and its complexes with $tRNA^{ser}$ were characterized by small-angle neutron scattering and contrast variation (EMBL and ILL). The enzyme alone has been crystallized, but the solution studies have established solvent conditions for complex formation as well as the geometry of the protein complex with two tRNA molecules.

The structures of viruses have for some time been studied by neutron scattering and recent experiments have focussed on understanding the assembly or disassembly of these particles. Welcoming Spain to membership of ILL, a collaboration was started with the Center of Molecular Biology in Madrid to study the interactions of viral connector protein of bacteriophage $\phi 29$ with DNA. Turnip Yellow Mosaic virus (TYMV) had been shown by electron microscopy to liberate its RNA genome on freezing and subsequent thawing. Neutron scattering experiments carried out on samples at 80 K have shown that the freezing process induces a swelling of the particles with a radial displacement of the RNA but that the RNA is liberated only on thawing (IBMC Strasbourg, ILL, EMBL). The thawed particles resemble closely those found in newly infected plants and the freezing/thawing process is thus perhaps similar to the *in vivo* decapsidation process. First experiments were carried out on virus-like particles (VLPs) from yeast. Genetic engineering techniques have been used to construct various forms of recombinant particle. Small-angle scattering is being used to determine the shape of the native

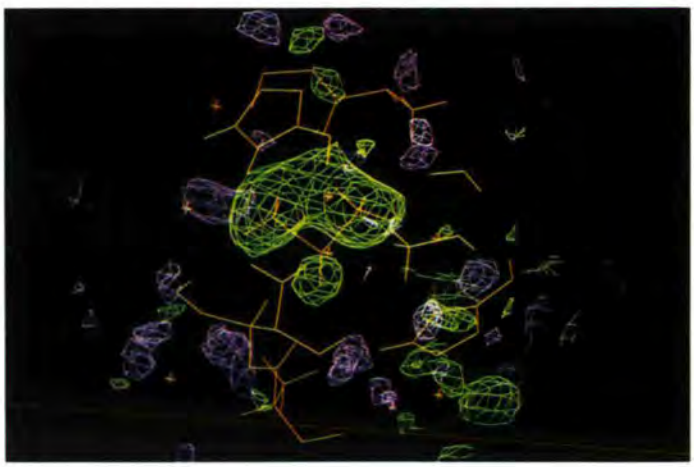


*Figure 73 Difference scattering density map showing a deuterated dimethyl sulfoxide molecule ($OS(CD_3)$) near the active site of lysozyme. Part of the protein skeleton is outlined.*

particle and of the recombinant forms (Oxford, EMBL, ILL). An important methodological advance has been made with the new triple isotopic replacement contrast variation method. This approach, proposed at the Institute of Protein Research (Poustchino, USSR), was tested experimentally at ILL on *E. Coli* EFTu and its interaction with tRNA, with very promising results.

## Natural and artificial membranes

Deuterium labelling and the high contrast of lipids in $D_2O$ make membranes ideal objects for study by neutron scattering. Experiments carried out on model systems include an investigation of the nature of the monolayer of water molecules hydrogen-bonded to the headgroup amino moitey of DOPE by deuteration of two specific sites, and the study of binary mixtures (DOPC and DOPE) to see the effect of the interaction between the two headgroups (which is of crucial importance in physiological membranes where these lipids are abundant) (Edinburgh, ILL). Phase transitions in systems formed by mixtures of phospholipids and fatty acids have also been examined (Southampton, ILL). Small-angle scattering from unoriented samples was used to determine the form and size of micelles as a function of the chain length and degree of saturation. In oriented samples deuterated fatty acid was used to locate this component with respect to the phospholipid (in the Lβ phase).

Model membranes under high pressure (up to 2000 bars) were studied for different head groups, chain length and degrees of chain saturation (Marburg, ILL). In addition to the liquid crystalline and gel phases a third phase appeared for DPPC by increasing the pressure, corresponding to a smaller lamellar spacing which was previously interpreted as an interdigitation of the chains. This study is a preliminary for understanding the molecular mechanism of the adaptation of bacterial and mammalian cell membranes to high pressure. In order to clarify results obtained by fluorescence experiments in lipid bilayers containing a fluorescent probe (DPH), neutron diffraction on oriented lipid bilayers containing the DPH molecule was performed. The localization of the DPH within the bilayer in the liquid crystalline and the gel phases was determined with deuterated and protonated DPH samples (NRCC, Bordeaux, ILL).

The major work on natural membranes is that carried out on the purple membrane of *H. Halobium*. The protein component of the membrane, bacteriorhodopsin, is a 26K molecular weight protein that functions as a light-driven proton pump. The path of the polypeptide chain in the low resolution structure of bacteriorhodopsin has been studied by neutron diffraction on specifically deuterated purple membrane samples (Institut de Biologie Physico-Chimique, Yale University, ILL). In the most recent experiments, an approach combining the deuteration of certain amino-acids and reconstitution of the functional molecule from two chymotryptic fragments has allowed the unambiguous assignement of two of the seven α-helices, in the primary structure, to specific locations in the low resolution map (Fig. 71). Further experiments have been carried out to locate the negatively charged C-terminus of the protein. Using enzymatically cleaved bacteriorhodopsin with Fourier difference techniques it has been shown to be located, in projection, close to the retinal chromophore (ILL) (Fig. 72). In the light, bacteriorhodpsin has a functional cycle of the order of milliseconds, in which its absorption spectrum changes through a number of well-defined states. By using special solvents and low temperatures, it has been possible to trap a specific state (the M-state) and study its structure and hydration (Freie Universität, Berlin and ILL). Small but reproducible structural differences have been observed between the M-state and the so-called dark-adapted state, the best characterized so far.

Experiments on detergent solubilized solutions of the photosystem I complex from *Synechococcus sp.* have shown that the active complex in the membrane is a monomer (Freiburg, ILL). Data have been measured on single crystals of the Reaction Center of *Sphearoides Rhodobacter* with the aim of localizing the detergent belt around the protein (Gif-sur-Yvette, ILL).

## Solvent interactions and protein stability

The work on the stability of proteins from extreme halophiles has continued on a halophilic GAPDH (BARC, India and ILL), a halophilic dihydrofolate reductase (Tel Aviv University, Weizmann Institute and ILL) and the halophilic EFTu (EMBL and ILL). From earlier neutron and complementary studies on halophilic malate dehydrogenase (hMDH), a novel structural model was proposed for this protein's thermodynamic stabilisation in its physiological saturated salt environment. The sequence of hMDH would constitute a useful test of the model structure. The gene has been cloned and the sequence determination is in progress (Tel Aviv, ILL, EMBL). Solvent interactions play a crucial role in the model, and its validity for other proteins that have adapted to a halophilic environment is now being tested.

The study of interactions between proteins and small solvent molecules is still a part of the activity in the college, and during the year diffraction data were recorded on an approximatley 60 $mm^3$ large triclinic crystal of hen egg-white lysozyme soaked in a 20% (v/v) solution of ethylene glycol. As in earlier similar studies the probe molecule was completely deuterated, while both the protein and the aqueous environment contained only hydrogen atoms.

The scattering density of water is thus nearly zero and only a few well-ordered water molecules are visible. On the contrary the solvent molecule is, due to the large scattering contribution of deuterium, easily observable, even when it is only partly present or disordered. Data were collected on D19 to a resolution of 1.9 Å, giving 7000 independent Bragg reflections. As usual the data are of good quality. At the present stage the crystallographic agreement factor from conventional structure refinement is 10.7%.

The analysis of recent measurements on lysozyme in 15% dimethylsulfoxide has been completed and shows several interesting features. As in earlier studies there is no detectable change at all in the structure of the protein molecule, neither concerning the atomic positions nor the thermal motions. A destruction of the structure, denaturation, is therefore more likely to pass via a dehydration at high solvent concentration rather than by a direct attack on the structure by solvent molecules. A comparison among the attachment sites for the

different solvent molecules studied (ethanol, dimethlsulfoxide and ethylene glycol) also shows similarities. They are in many cases the same for the three molecules and are mainly of hydrophobic nature. Of special interest is the active site of the enzyme, where we always find a solvent molecule bound to part of the wall. Fig. 73 shows the case for dimethyl sulfoxide. This binding is undoubtedly an essential part of the attachment of the substrate to the macromolecule during the enzymatic reaction.

## Inelastic scattering

The study of the dynamics of purple membrane as a function of temperature and hydration has continued on IN6 (ILL, EMBL). Samples specifically deuterated in the membrane lipids have been prepared in order to seperate the lipid and protein contributions to the scattering. The data obtained so far provide a frequency distribution of movement within the protein which will be compared with that predicted by computer simulation. A study of the inelastic scattering as a function of temperature (4 to 350 K) has been carried out on myoglobin. The dynamical behaviour found has many of the features expected for a liquid-glass transition and appears to be general for globular proteins (EMBL, Munich, ILL).

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# Molecular Spectroscopy, Surfaces and Mesophases

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## Scientific Highlights in 1988

### T-dependence of quantum mechanical motion

Quasi-elastic and inelastic incoherent neutron scattering has been used to study in detail the transition from quantum mechanical tunnelling motion to classical reorientation of the methyl groups in $CH_3CONH_2$ (ILL, Jülich). The temperature dependence of the low-temperature quasi-elastic and inelastic scattering due to $E_AE_B$ and AE transitions of the tunnelling methyl groups has been investigated between 4 and 200 K on IN13 and IN10 and together with the higher temperature quasi-elastic scattering, compared with theoretical predictions. An Arrhenius plot of the line shift and broadening of the AE tunnelling transition and of the line width of the $E_AE_B$ transition is shown in Fig. 74, displayed as well is the behaviour of the classical quasi-elastic peak at higher temperatures.

As expected the AE peak shifts to lower energies with increasing temperature, both tunnelling transitions broaden. Starting from about 30 K on a separation of the two components is no longer possible by data fitting, they merge into a common quas-ielastic peak. Activation energies and pre-exponential factors for the processes were determined. For the low-temperature range perturbation theory, taking into account the coupling between the rotor and the phonons correctly up to second order predicts that the line shift of the AE transition proceeds more slowly on temperature increase than the accompanying broadening. The activation energies for the broadening of the AE and $E_AE_B$ transitions should be the same and equal to $E_{01}$, the energy of the first librational level. These predictions can now be affirmed from the experimental results, the fact that the broadening of the $E_AE_B$ transition is only about

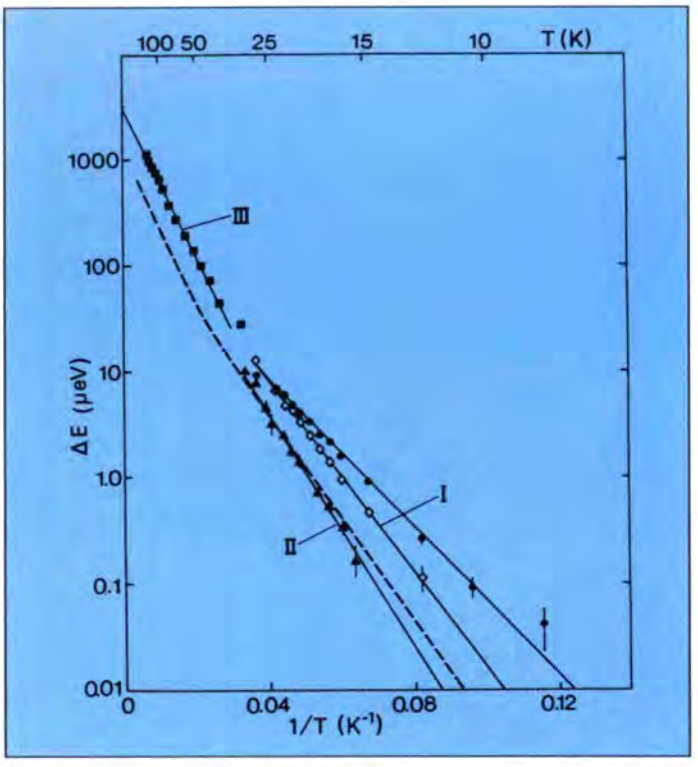
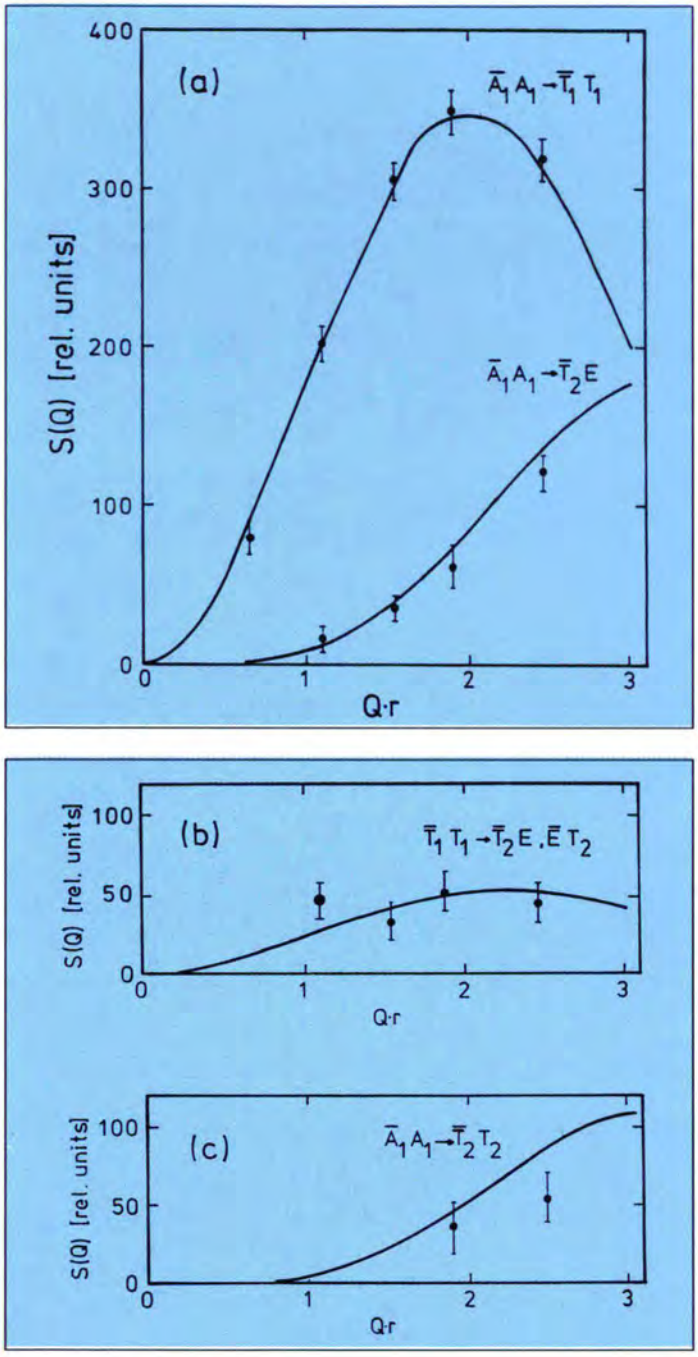



*Figure 74 Arrhenius plot of the line shift of the AE transition (●) and of the line broadening of :*
*I the AE transition (◊)*
*II the $E_AE_B$ transition (▲) and*
*III the "high" temperature quasi-elastic peak (■).*

half that of the AE transition could, however, not be explained up to now by theories which predict a similar size for both. A novel theoretical approach (Erlangen, ILL) to explain the temperature dependence of rotational tunnelling which was presented only shortly after this experiment had taken place and which was in fact stimulated by the experimental results seems, however, not only capable to solve the aforementioned problem but seems as well to describe correctly the behaviour of the quasi-elastic peak in the whole transition range and in the high temperature limit.

### Matrix isolation spectroscopy

Another experiment, which was devoted to the experimental verification of predictions made from theory, looked at the rotational excitations of $CH_4$-molecules matrix isolated in Argon (Jülich, Kiel, ILL). Measurements on a sample containing 0.3% $CH_4$ were performed at different momentum transfers on IN3. Four INS peaks are in comparison with the energy level scheme as developed for solid $CH_4$-II and from their absolute values attributed to the E(0-1), E(0-2), E(1-2) and E(0-3) transitions. The rotational levels are as a consequence of the stronger crystalline field in the Argon matrix lower than in solid $CH_4$-II Much more information is, however, contained in the Q-dependence of the scattered intensities. The transition probabilities as calculated for $CH_4$-II on the basis of an 'Extended James-Keenan Model' can be found in the literature. Knowing their Q-dependence, it is possible to evaluate the double differential scattering function




*Figures 75 & 76*
*Inelastically scattered intensities of matrix isolated $CH_4$. The solid lines refer to least-square fits according to the theoretical model developed for solid $CH_4$-II. The C-H distance in a $CH_4$-molecule is 1.093 Å.*

for the four transitions seen in the INS-spectra. Figs 75 and 76 show that the scattered intensities as measured for the four peaks can be fitted successfully with the theoretical model, confirming the unambiguous assignment of the different transitions. Comparing the two phases $CH_4$ matrix isolated in Argon and solid $CH_4$-II it can be stated that the stronger crystalline field in the first phase seems mainly to influence the rotational transition energies but only slightly the corresponding transition matrix elements.

The large number of bands in the IR spectrum of **urea isolated in an Argon matrix** has been suggested to be due to inversion tunnelling of the $NH_2$ groups. This situation is particularly interesting because there are two tunnelling groups in the molecule and only simultaneous tunnelling by both groups results in a configuration of unchanged energy. Accordingly, a large dilute matrix for INS was prepared, containing 3 mg of urea in 28 g of Argon (ILL, East Anglia).

The INS spectra (IN5) of this matrix revealed main bands at 24, 18 and 4 $cm^{-1}$ which were not seen in the spectrum of pure Argon and were also found to disappear after annealing the sample at 60 K for 1 hour. The Hamiltonian can be constructed by designating the nitrogen atoms "a" and "b" with conformations U and D.

| | $U_aU_b$ | $D_aD_b$ | $U_aD_b$ | $D_aU_b$ |
|---|---|---|---|---|
| $U_aU_b$ | -S-E | $t_2$ | $t_1$ | $t_1$ |
| $D_aD_b$ | $t_2$ | -S-E | $t_1$ | $t_1$ |
| $U_aD_b$ | $t_1$ | $t_1$ | +S-E | $t_2$ |
| $D_aU_b$ | $t_1$ | $t_1$ | $t_2$ | +S-E |

Products of the eigenfunctions of this matrix with the spin functions allow the intensities of the transitions to be calculated (inset in Fig. 77). From the spectrum, the three parameters of the Hamiltonian are found to be: $2S = 13$ $cm^{-1}$, $t_1 = 5$ $cm^{-1}$ and $t_2 = 0.5$ $cm^{-1}$, the energy level scheme being shown in Fig. 77. The dominant peak in the spectrum is assigned to the $^5A \rightarrow {}^5A$ transition, with the weaker feature at 18 $cm^{-1}$ being $A \rightarrow B_3$. The very weak band at 4 $cm^{-1}$ then fits the $A \rightarrow B_2$ assignment well. This assignment is indicated by the vertical arrows in Fig. 77.

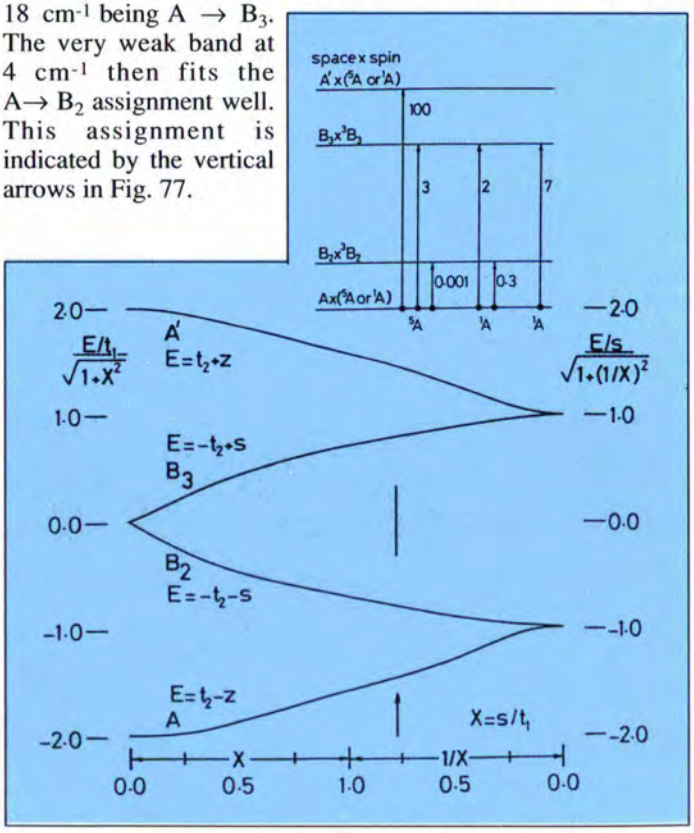



*Figure 77 Energy level scheme for urea [$z = (S^2 + 4t_1^2)^{1/2}$], the inset shows the relative intensities for the transitions.*

## Tunnelling in a molecular glass

Following the recent work on transition metal hexammine complexes (see annual report 1987) it was of particular interest to study the low temperature/low energy rotational excitations of the $NH_3$-groups in $Ca(NH_3)_6$, as one example of several alkaline earth metal complexes with ammonia (Lille, Kiel, ILL). The bcc structure of this complex is very open, the volume expansion at formation is 45%. At high temperature the $NH_3$-groups undergo rapid rotational diffusion which stops at 30 K. The structure remains bcc down to 1.5 K where the $NH_3$-groups freeze in orientational disorder (molecular glass).

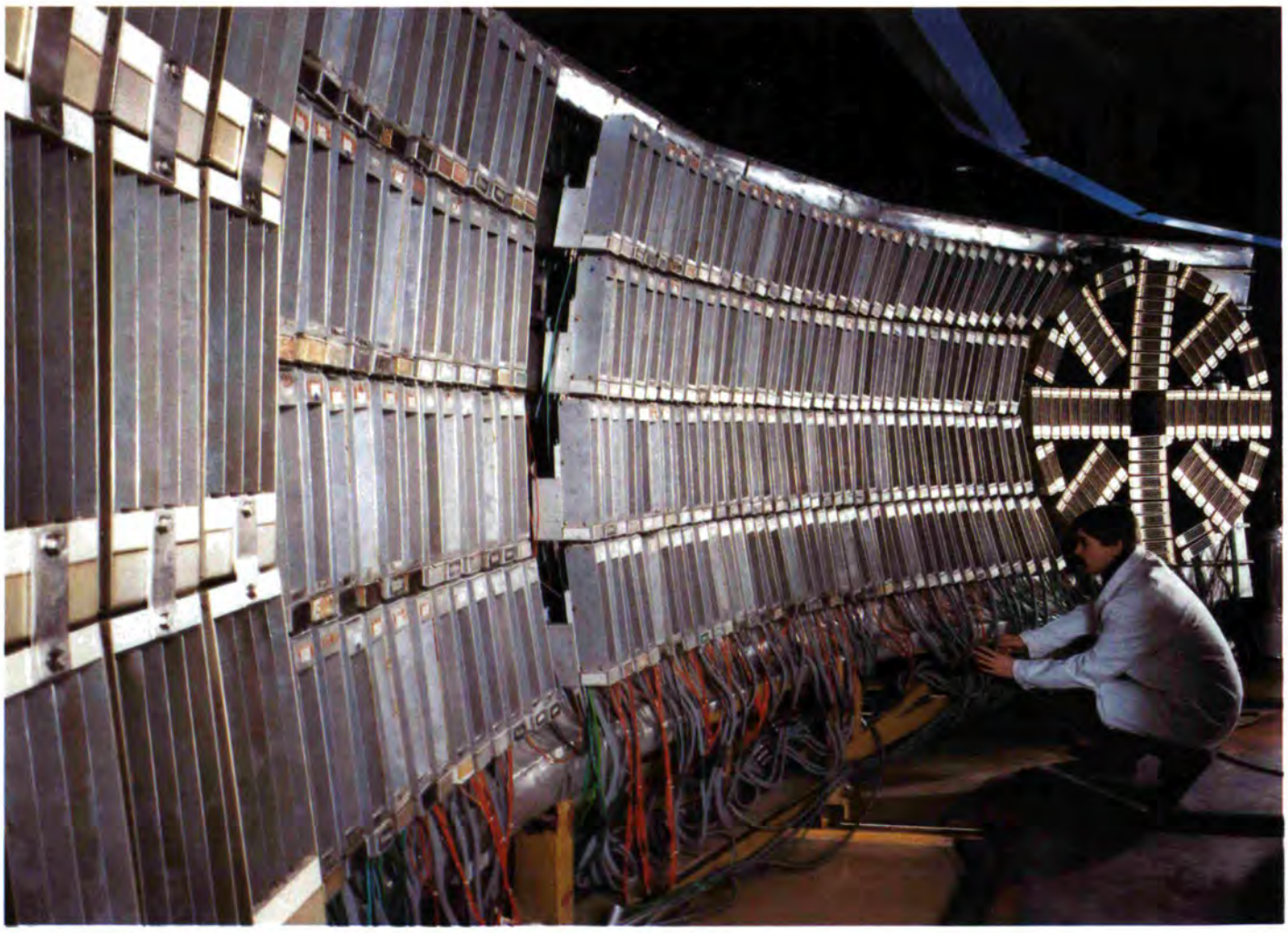
As a finger print of the disorder the proton tunnelling observed at low temperatures does not show up as a well defined transition energy but as a broad peak, reflecting a continuous distribution of hindering potentials going from free rotation to barrier heights of several meV.

Looking at the series $Ca(NH_3)_n$ (n = 5.5, 6.0, 6.5) the first rotational tunnelling transition was used to determine the density of states serving as local probe to study the disordered materials (Fig. 78).

Looking at the compound with n = 6 it was striking to find the inelastic intensity cut down at the very free rotor energy (73 meV as determined from the structure). This is probably the first proven evidence of a free uniaxial rotor at low temperatures. The density of states g(E) was calculated (Fig. 79), most of the states are close to the free rotor. Removing ammonia from the system (n = 5.5) leads to an increase of the average barrier height, the density of states near the free rotor remains nevertheless rather high. The situation is, however, different for n > 6. The inelastic scattering for the 0 to 1 transition has significantly decreased (35% of that found for n = 6.0), the number of states near free rotation is very low (Fig. 80). A sharp peak is visible at 60 μeV and some structure appears in the distribution. It seems that the additional $NH_3$-groups go on interstitial sites, blocking most of the motion.

## Electron transfer seen by INS

Electron transfer processes are of fundamental importance in fields ranging from biology through chemistry to solid state physics. A central problem is to measure the rates of electron transfer between metal ions in crystallographically well-defined sites, and for this purpose **tri-nuclear metal cluster complexes** of the type shown in Fig. 81 are particularly useful model systems. They can be prepared with all 3 metal ions identical, with mixed clusters of two different metals, or with



*The array of $^3$He detectors on IN5.*

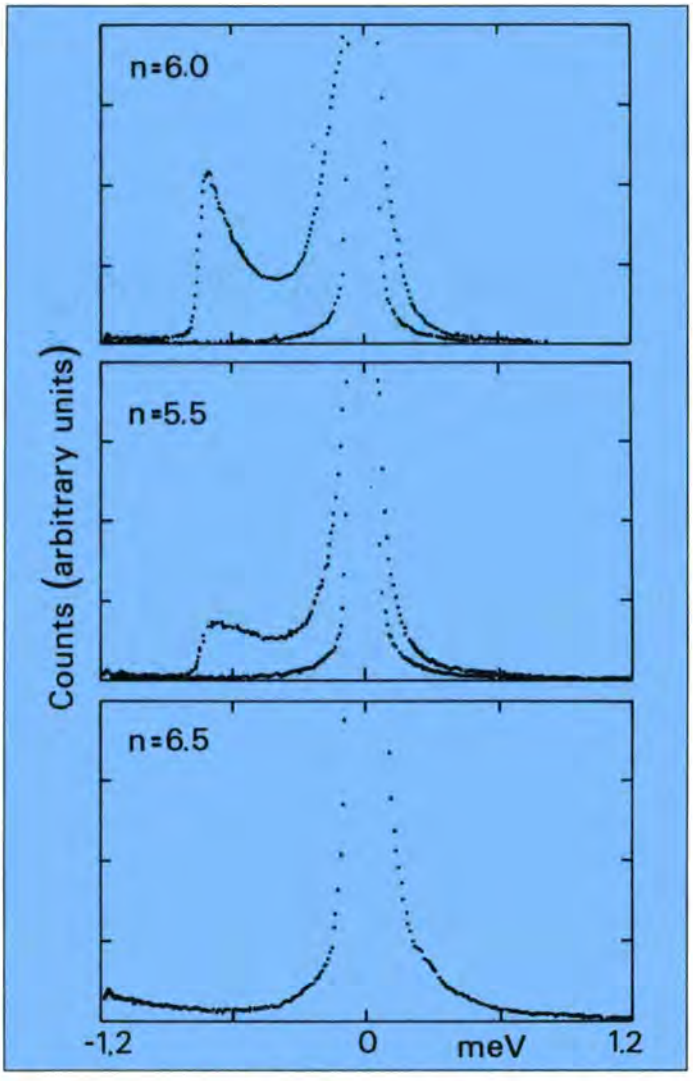



*Figure 78 First excitation (0→1 transition) of the uniaxial rotation of $NH_3$ groups in $Ca(NH_3)_6$ with n = 6.0, 5.5 and 6.5 respectively. Each spectrum was scaled at 5% of the elastic peak. The elastic line observed with a vanadium sample has been superimposed to indicate the spectrometer resolution (Vector transfer $\overline{Q}$ = 1.5 Å$^{-1}$).*

mixed-valence clusters of the same metal. Thus we have a unique opportunity to compare the strength of electronic interactions between various metal ions under closely similar conditions.

**Inelastic neutron scattering was used to measure the strength of the intermetallic exchange interaction** in a number of homologues of this series (ILL, East Anglia). So far, the results have been interpreted in terms of a simple isotropic coupling mechanism as shown for the tri-chromium (III) system (Fig. 82a). In general the results reinforce those already obtained from magnetic susceptibility studies, but with the advantage of direct spectroscopic observation of temperature dependent effects (a weakening of the magnetic coupling on increasing temperature is discernable in Fig. 82b).

For the mixed valence complexes where there is the possibility of electron delocalization between the metal centres, raising the temperature from 5 to 50 K is accompanied by a dramatic band broadening. This has not been observed for the heterometallic or homovalence systems studied thus far. The exact origin of the broadening is, at present, uncertain, but is believed to be due to a static delocalization of electronic charge over the metal triangle.

Intramolecular electronic interactions have been studied as well on IN6 looking at a **new class of compounds, molecular hydrogen complexes**, which for the first time exhibit direct coordination of a σ-bond to a metal. This provides a unique opportunity to study intramolecular interactions by means of rotational tunnelling spectroscopy. Work at the ILL (Los Alamos, ILL, Univ. of Toronto) has shown that rotational tunnelling by the $H_2$-ligand can be described by planar rotation in a double minimum potential, and that the barrier to rotation is determined to a significant extend by direct electronic interaction with the metal. The two-fold symmetry apparently arises from the backdonation of electrons from a metal d-orbital to the σ* orbital of the $H_2$. Recent studies involving Mo and Fe complexes with chelating diphenylphosphinoethane ligands have focussed on the effect of the ligand **trans** to the

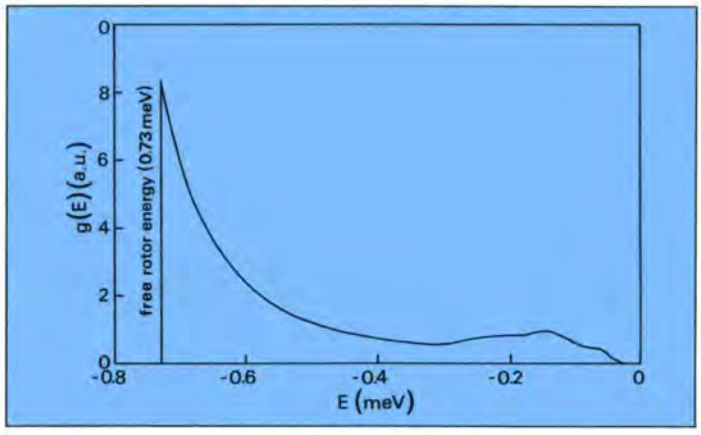



*Figure 79 Normalized density of states g(E) in arbitrary units for the $Ca(NH_3)_{6.0}$ compound at 1.4 K. Most of the states are located near free rotation $\Delta E = \hbar\omega/2I$ = 0.73 meV.*

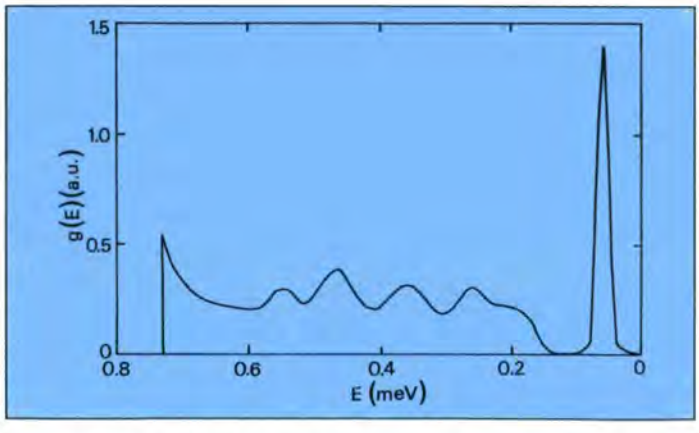



*Figure 80 Normalized density of states g(E) in arbitrary units for the $Ca(NH_3)_{6.5}$ compound at 1.5 K.*

$H_2$ (e.g. a hydride or carbonyl group). These have pronounced effects on the electron flow between the metal and the $H_2$ ligand which is then reflected in the rotational barrier. It is the sensitivity of rotational tunnelling which makes these studies successful : so far, observed tunnelling frequencies in these systems have covered a range of a factor 80 in energy.

## Surface studies

QENS was used to study the surface premelting of thin films of methane adsorbed on MgO (ILL-Marseille). At low temperatures the whole $CH_4$-film is solid, the line width of the INS-spectra is resolution limited. From 72 K onwards (i.e. about 18 K below the $CH_4$ bulk melting point), a broadening of the spectra is observed. The spectra were analyzed with a two-term scattering law, taking into account the simultaneous existence of a solid and a liquid fraction. From the relative intensity of the wider component of the QENS spectra the quasi-liquid thickness could be calculated. It changes from ~ 1 layer at 72 to ~ 5.7 layers at 90.3 K. The Lorentzian function representing the incoherent scattering by the mobile phase is model-dependent, a good fit was obtained for the spectra below 87 K when choosing for the geometrical factor entering:

$$f(Q) = [2\text{-}\cos(\mathbf{Q.a})\text{-}\cos(\mathbf{Q.b})]/2t$$

which is valid for a square lattice fluid. Above 87 K the model of a two-dimensional lattice fluid where the $CH_4$-molecules jump between equi-distant lattice sites becomes less appropriate to describe the now several layers thick liquid phase.

In the limit of small Q f(Q) provides directly the translational diffusion coefficient D. It was shown that the surface mobility is about twice that of bulk liquid in the vicinity of the melting point. Assuming a motion of the bulk liquid type (as well perpendicular to the surface) the diffusion coefficient is reduced by a factor of 1.5 but still larger than that of the "true" liquid. The high value of d in the premelting layers is probably related to the large concentration of vacancy adatom pairs.

The behaviour of the **quantum gases hydrogen ($H_2$) and its isotopes, deuterium ($D_2$) and deuterated hydrogen (HD)**, is of considerable experimental and theoretical interest, in particular for the study of their phase transitions in two dimensions (2D). These gases adsorbed on graphite are a good approach to an ideal 2D system in the incommensurate phase, but lock at lower coverages into a ($\sqrt{3}$ x $\sqrt{3}$) R30° commensurate phase (C-phase) due to the graphite surface potential corrugation.

The out-of-plane oscillation of the adsorbed molecules is given by the variation of the adsorption potential in the direction normal to the graphite surface plane. The in-plane oscillations in the C-phase are given by the corrugation of this potential along the surface and the interaction between the adsorbed molecules.

The parameters which characterize the dynamics of the three isotopes in the C-phase were obtained from data taken with the triple-axis spectrometer IN3. They are given in the table together with theoretical values found in the literature. A good overall agreement between experiment and theory is found and the DOS shows the expected trend for the different isotopes. Unfortunately there are not yet calculations for HD available. The zone centre gap, which reflects the corrugation of the substrate, is found at a somewhat higher energy for both species, but the agreement is very reasonable. A significant point is that the width of the DOS is overestimated in the calculations (the theoretical values are 50% too high for $H_2$ and $D_2$). This implies that the interaction between the adsorbed molecules is in fact weaker than expected. The values of the phonon gap and the width of the DOS do not scale with inverse

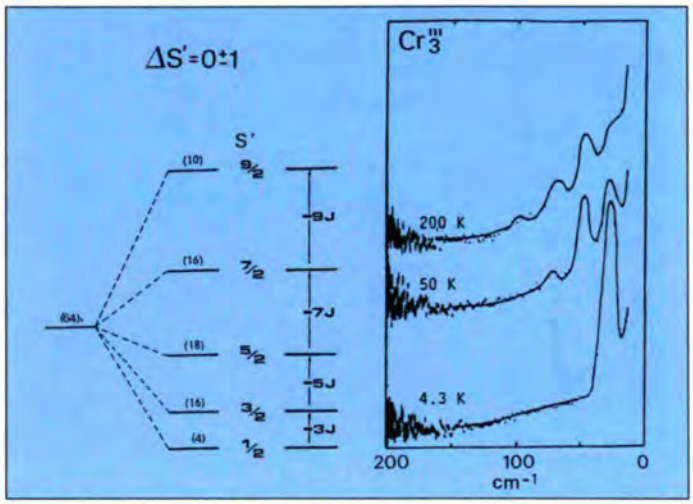



*Figure 82 Coupling of a symmetrical triad of high spin S = 3/2 ions give rise to the IN4 spectrum on the right.*

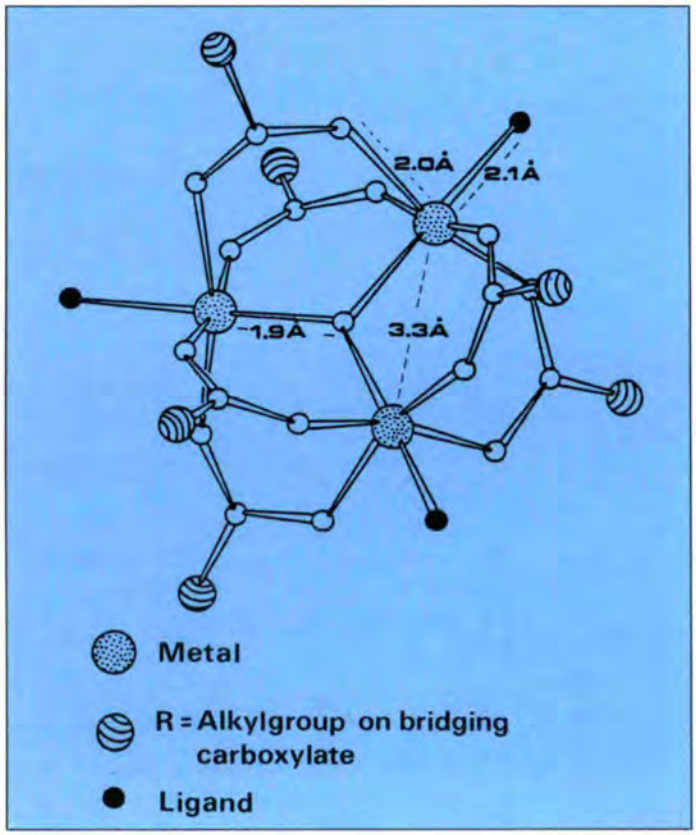



*Figure 81 Structural unit of the trimetallic oxo-centred complexes.*

of the square root of the mass of the isotopes as expected for a classical system. $H_2$ which has a larger zero point motion interacts more strongly with his neighbours than HD and $D_2$. This is evidenced by the width of the DOS which is for $H_2$ nearly three times that of $D_2$, instead of being $\sqrt{2}$ due to the isotope effect.

At the same time the corrugation of the substrate is averaged out due to the zero point motion and thus the gap for $H_2$ lies much lower than what one would expect considering only the isotopic shift.

| | H2 | HD | D2 |
|---|---|---|---|
| z. c. gap | 47.3 (46.6) | 43.2 | 40.0 (36.9) |
| | [49.7] | [40.5] | [36.2] |
| width | 27.5 (42.1) | 14.7 | 9.5 (14.8) |
| trans. peak | 57.9 (64.9) | 48.8 | 43.3 (44.2) |
| long. peak | 71.4 (83.8) | 55.8 | 48.1 (50.3) |

*Parameters obtained from the fits characterizing the density of states for the in-plane modes of the ($\sqrt{3}$ x $\sqrt{3}$) R30° commensurate phase of the hydrogen isotopes adsorbed on graphite (in parenthesis the theoretical values and between brackets the frequencies for the Einstein-mode obtained with a classical adsorption potential). Indicated are the following: z.c.gap is the zone centre gap energy; width is the DOS width; tran. peak and long. peak are the peaks arising from the transverse and longitudinal phonons in the DOS, respectively (all values are in Kelvin).*

## Dynamics of inclusion compounds

Nitric acid molecules intercalated in graphite exhibit a continuous melting behaviour above 247 K. The incoherent quasi-elastic scattering from an oriented specimen was investigated using the spin-echo spectrometer IN11. The results (especially the EISF versus Q) show that the molecular diffusion in the liquid phase is typical of that of a lattice liquid (hopping diffusion on the graphite potential), but within a restricted area of about 20 Å diameter (no long-range diffusion). Measurements in the vicinity of the transition temperature show a critical decrease of the residence time together with a decrease of the elastic scattering (Fig. 83 shows the results between 253 K and 257 K). This is interpreted as due to the proliferation of pairs of dislocations. The melting itself occurs only at 257 K when the interdistance between pairs reaches their typical size. Such a behaviour is different from that predicted by Halperin and Nelson in the model of "dislocation mediated 2-D melting" according to which the l-melting occurs by separation of the two dislocations of each pair (Fig. 83).

The motional behaviour of **hexadecane - $h_{34}$ within the pseudo-hexagonal channels provided by the urea-$d_4$ framework** has been studied by incoherent quasielastic scattering between 5 K and 370 K (München, ILL). On the instrument time-scale (80 μeV), at least two components could be separated, which are attributed by fitting their Q-dependence to reorientational jumps of the paraffins around their long axes and damped librations, respectively. At 147 K a phase transition takes place involving an orthorhombic deformation of the host channels and a simultaneous change of 60° to 180° jumps. From the activation energy, it is concluded that the motional properties of the included molecules are responsible for this transition.

Secretary: C. Ritter

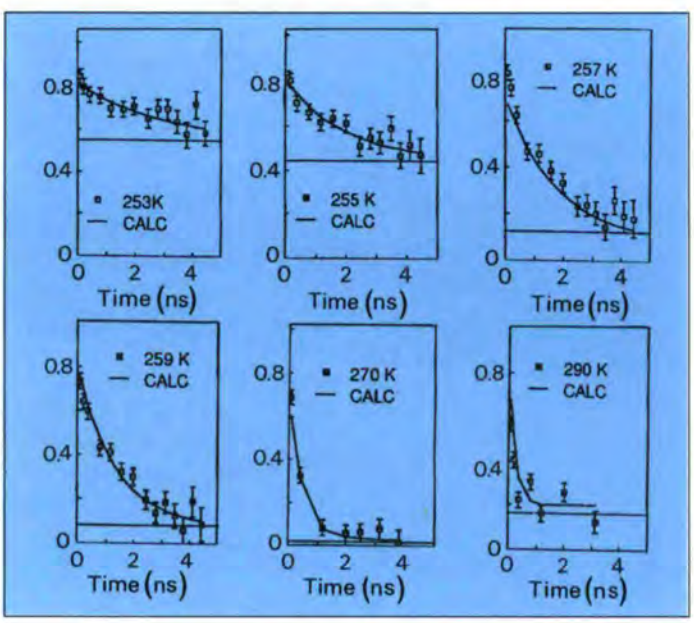



*Figure 83 Intermediate scattering function S(Q,t) at Q = 0.54 Å$^{-1}$ (incoherent scattering in the (hk0) plane from $HNO_3$ intercalated graphite).*

# Phases and Phase Transitions of $D_2$ on Graphite Obtained by Variable $^4He$ Pressure

V. L. P. Frank, H. J. Lauter and P. Leiderer

Phases and phase transitions of $D_2$ on graphite have been investigated using $^4He$ as 2-dimensional pushing-gas. The two gases seem not to mix. By this unique technique, density dependent phase transitions have been studied along a quite unusual path in the $D_2$ phase diagram.

The phase diagram of a monolayer of $D_2$ on graphite is a typical example of a quantum gas on graphite. The transition from the ($\sqrt{3}$ x $\sqrt{3}$) R30° commensurate phase (C-phase) to the incommensurate phase as a function of coverage exhibits a couple of intermediate phases (Fig. 84). The phase diagram has been defined by heat capacity measurements[1].

Here we want to concentrate on the C-, the α- and the ß-phase which are shown for the pure $D_2$-graphite system in Fig. 84. The diffraction peaks observed in the α-phase have been fitted with a model of super heavy striped domain walls[2]. The ß-phase has a liquid-like structure factor which is modelled by small pieces of domain walls, because the same excitations as seen in the α-phase are maintained at least near the α-ß transition[3] (in particular the transverse phonon which is characteristic of a solid phase). The dashed line between the C-phase and ß-phase in Fig. 84 is just a guide to the eye connecting the high heat capacity peaks of the melting C-phase to the start of the α-ß transition.

In order to investigate the phase transitions as a function of pressure another gas can be co-adsorbed and, provided both gases do not mix, it can act as pushing gas (2-dimensional (2D) piston), driving the host gas through phase transitions as a function of spreading pressure or density. This effect is analogous to a coverage dependence for a pure adsorbate.

In our case we have used $^4He$ as pushing gas. It can adsorb or desorb without difficulty at a constant temperature e.g. T = 6 K. In this experiment, however, we added the $^4He$ at a temperature where the $D_2$ is adsorbed in the fluid phase and cooled the system. During cooling down the $D_2$ becomes solid first, but also the $^4He$ adsorbs and increases the 2D spreading pressure, thereby compressing the $D_2$. The vapor pressure of the 3-dimensional $^4He$ gas was recorded down to T = 5 K. From a series of $^4He$ adsorption isotherms the 2D spreading pressure was calculated[4] (Fig. 85).

The neutron diffraction scans shown in Fig. 85 have been taken on the D16 spectrometer at the ILL on the cold source at a wavelength of 4.526 Å. The sample consisted of a stack of ZYX-graphite. The coverage of a complete commensurate layer was 14.43 $cm^3$ STP (coverage ρ = 1).

The runs in Fig. 85 show the diffraction of the $D_2$ because the neutron cross section of $^4He$ is nearly 20 times smaller than the one of $D_2$. A first filling of 11.1 $cm^3$ STP $D_2$ was made. Then an amount of 8.0 $cm^3$ STP $^4He$ was added. The whole system was thermalized at 38 K and slowly cooled down.

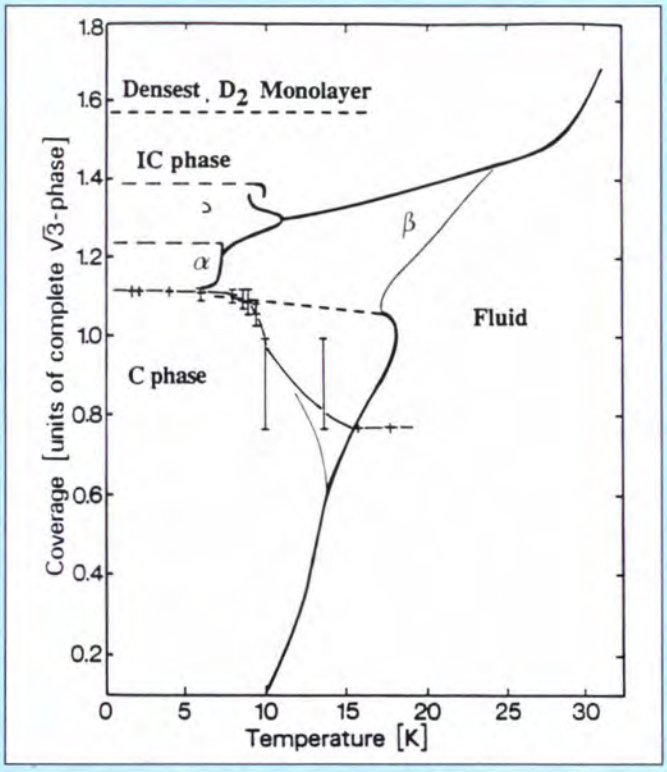



*Figure 84 Monolayer phase diagram of $D_2$ on graphite. The phase transitions (solid line) are given by heat capacity measurements[1]. The dashed line is a supposed transition between C- and β-phase and is discussed in the text. The meaning of C-, α-, β-phase is also explained in the text. The line through the points indicates the path in the phase diagram of $D_2$ on graphite accomplished by compression due to the co-adsorbed $^4He$.*

For each neutron scan the system was kept at a constant temperature. By this technique different phases are crossed (see Fig. 84). In order to draw the points of the followed path into the phase diagram of pure $D_2$ we used as definition of the coverage the deduced density of the $D_2$ alone (explained in more detail in Ref. 5):

- The two spectra, taken at 16.9 K and 15.9 K, exhibit liquid character of the $D_2$ alone, because $^4He$ is assumed to be in the second layer and above.
- At 13.9 K the lattice spacing (at Q = 1.703 $Å^{-1}$ is the (10) peak of the triangular structure) is the one of the C-phase. The same holds for the next spectrum taken at 10.0 K. Here the peak intensity has increased because the Debye-Waller factor increased. For T < 10.0 K the peak intensity drops and the width of the peak increases due to an effective density ρ > 1 of the $D_2$ (the overfilling of the C-phase creates interstitials).
- At 8.0 K a shift of the peak position indicates the transition to the ß-phase (This may define the dashed line in Fig. 84 which is not seen in heat capacity measurements). At 6.0 K a further shift of the peak position is noticeable but the line shape still indicates the ß-phase. Thus this point must be placed just below the transition line between the α- and ß-phase.

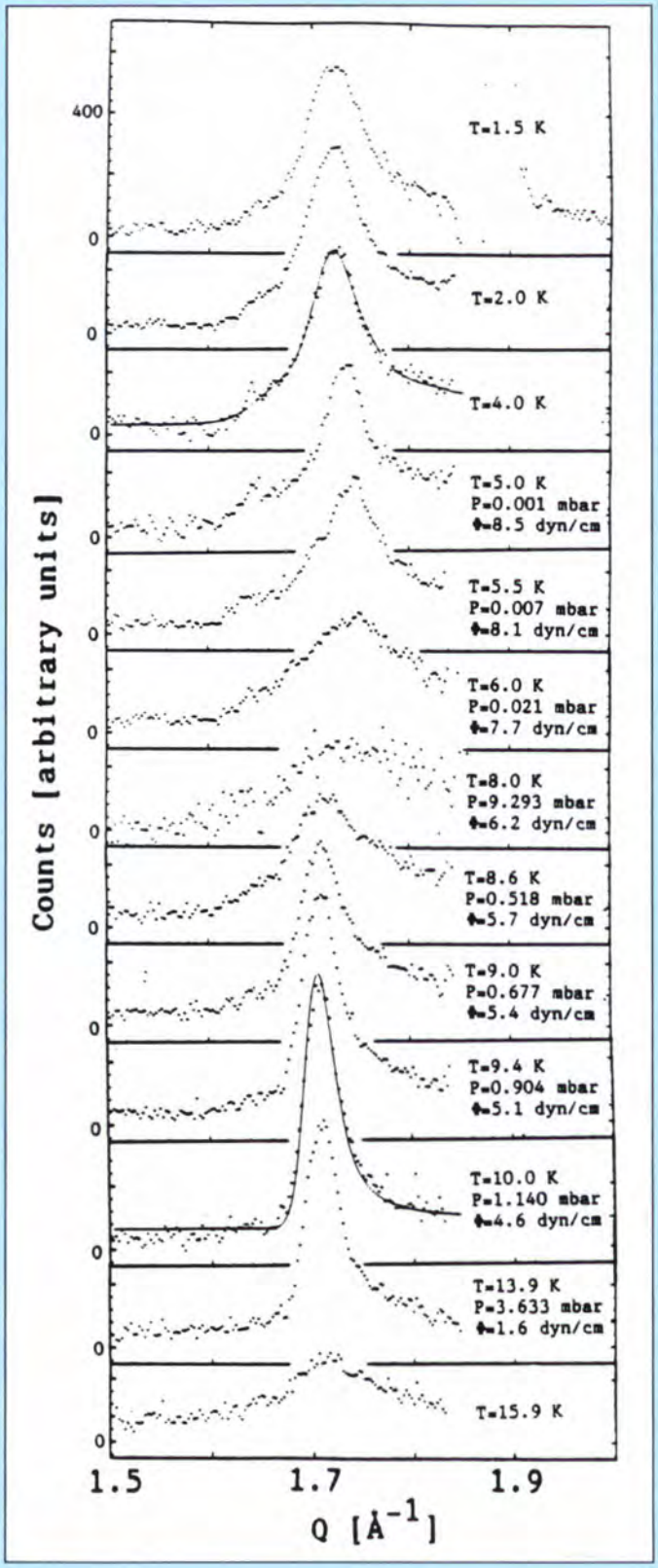



*Figure 85 Sequence of neutron diffraction spectra as a function of temperature. The temperature at which each diffraction spectrum was taken, the measured 3-dimensional $^4$He pressure and the calculated spreading pressure[4] are marked.*

- Further cooling brings about a decrease in the width of the peak and the first appearence of satellites. The scan at 4 K shows that the line shape can be well fitted with the model of a Monte-Carlo distribution of superheavy domain walls of the pure $D_2$-graphite system[2]. This fit gives a value of $\rho = 1.116$ at which this scan is marked in Fig. 84. This is in agreement with the coverage-position of the scans at higher temperatures. It should be pointed out that the line shape is identical to the one calculated for the pure $D_2$ system. Thus the $^4$He has no influence on the structure factor of the $D_2$ lattice and consequently we argue that it does not mix with the $D_2$.
- It is interesting to note that the spectra at 5.0 K and 5.5 K display a new kind of line shape which cannot be fitted up to now. New satellites appear between the main peak and the satellite position of the α-phase.

The 2D spreading pressure can be used to estimate the relaxation of a $D_2$-molecule in a superheavy domain wall (relaxation with respect to the adsorption site on the graphite [3]) neglecting the interaction with the $D_2$-molecules next to the domain wall. A value of 0.2 Å is obtained if a spring force constant of 91 dyn/cm[6] (in-plane between $D_2$ and graphite) is taken. This value has to be compared with the one of 0.68 Å resulting from the calculation of the structure factor in the domain wall phase [2]. The two values have the same order of magnitude and the difference can be explained by the fact that the force constant taken is the one at the adsorption site and not the one at the relaxed position. The agreement between the two values of the relaxation deduced from two different experiments is reasonable.

To conclude, we have shown for the first time that a compression of the $D_2$ monolayer on graphite by co-adsorption of $^4$He is possible. This permits to study delicate parts of the $D_2$ phases and its phase transitions and to get more valuable information about the domain walls. Here we presented a study which includes the transition from the commensurate phase to the domain wall liquid phase and from this phase to the striped superheavy domain wall phase.

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# Colloids and Polymers

### Members of the College

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A. Wright

## Introduction

Some general remarks should elucidate the situation of the research activities of College 9b in 1988. The total number of submitted proposals dropped from 85 (Scientific Council October 1987) and 93 (March 1988) to 59 in October 1988 which could only partly be explained with the fact that proposals concerned with reflection experiments are discussed in subcommittee 9a since October 1988. Fig. 86 shows the distribution of accepted experiments for the different instruments according to the subcommittee decisions of the Scientific Councils in October 1987, March 1988 and October 1988. As in previous years, most of the experiments were allocated in the field of small angle scattering at the instruments D11 and D17 (with an average beam-time allocation of 1 - 2 days). Due to the breakdown of the D17 multidetector in October 1988 (an estimate for the date of availability for the repaired detector is early spring 1989) the experiments of the last reactor cycle on this instrument had to be postponed. Some of the experiments proposed for D17 at the Scientific Council in October 1988 had been shifted to D11 which led once again to a heavy overload on this instrument.

The distribution of the diverse topics in the College during this one-year period is illustrated in Fig. 87a,b,c. Most of the accepted experiments are concerned with the subject colloids (subject keywords 09 and 10), also contributing with the largest fraction of "new" proposals, followed by experiments in the field of polymers in solution, melts, amorphous polymers (keyword 11) and semi crystalline, liquid crystalline polymers (keyword 13).

Another feature is the national distribution which is illustrated in Fig.88. It shows that collaboration between the major member countries (France, Germany, UK) seems to be restricted to some exceptional cases. The collaboration of the College with groups from the member countries varied between 14% (March 1988) and 30% (October 1988). Spain (member country since 1987) contributes with about 3% to the experimental work.

The few selected examples given below illustrate some of the various scientific activities of College 9b.

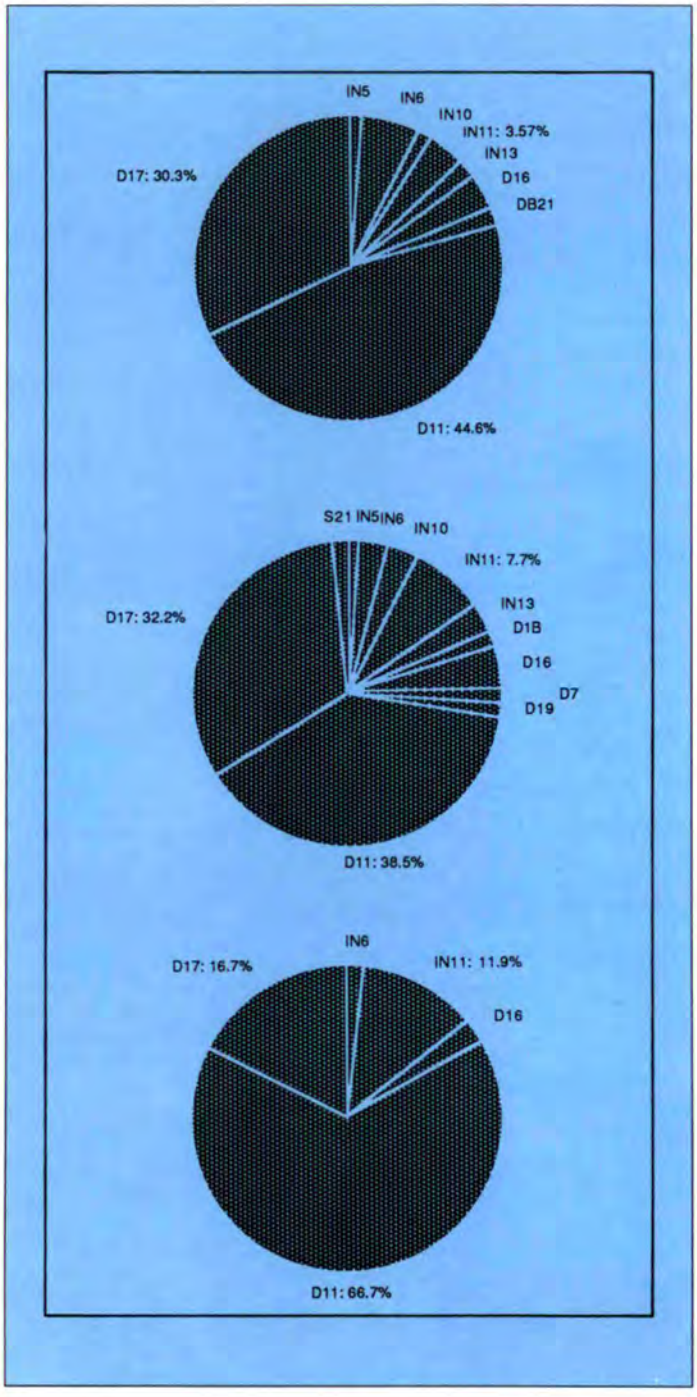


*Figure 86 Percentage distribution of accepted proposals by instrument in College 9b.*
*a) Scientific Council October 1987,*
*b) March 1988,*
*c) October 1988.*

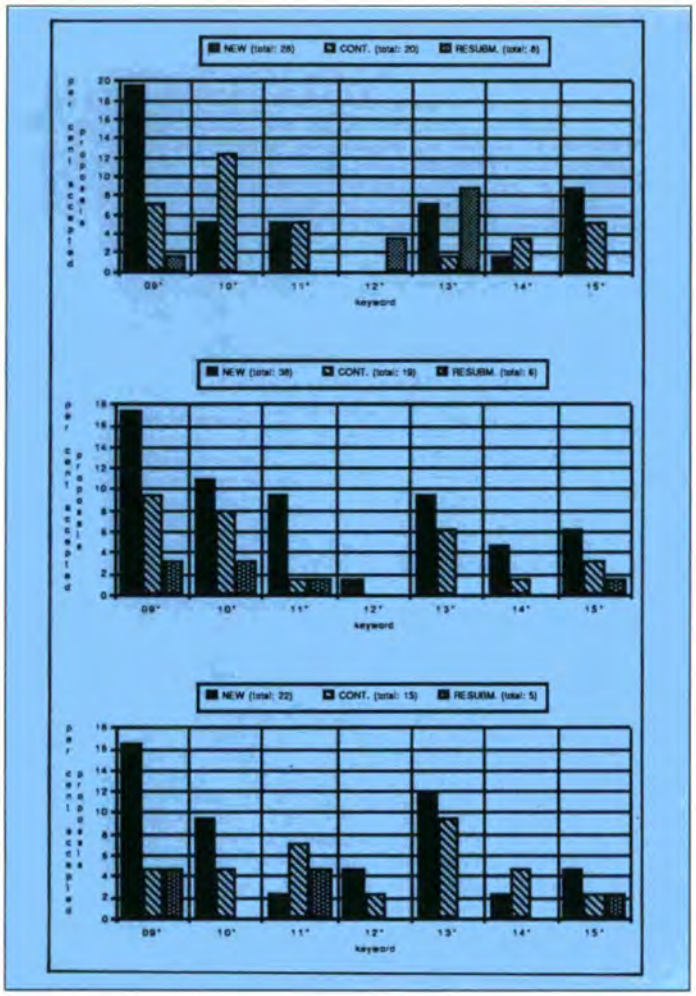

*Figure 87 Percentage distribution of accepted proposals by subject in College 9b. a) Scientific Council October 1987, b) March 1988, c) October 1988*

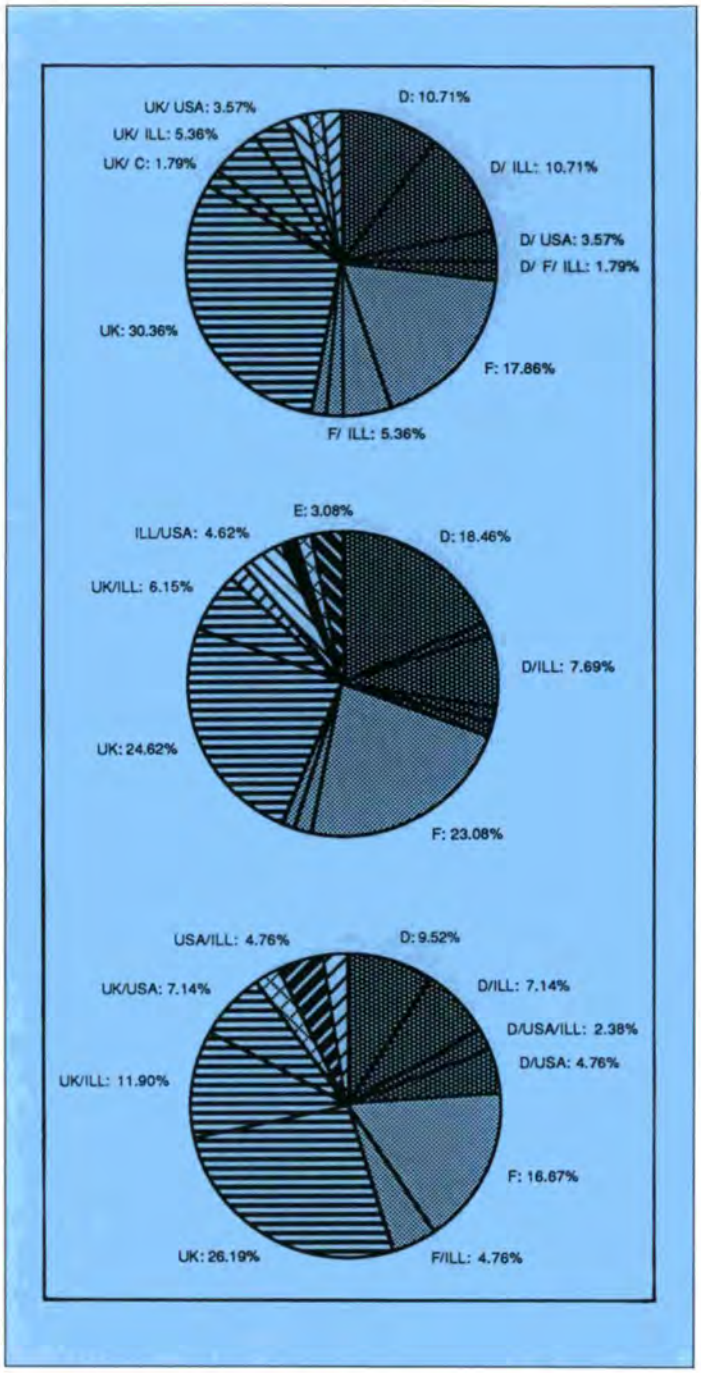


*Figure 88 Percentage distribution of accepted proposals on different countries in College 9b. a) Scientific Council October 1987, b) March 1988, c) October 1988.*

# Scientific Highlights in 1988

## Organogels

The investigation of surfactant aggregation in organic media becomes of considerable fundamental and applied interest in the fields of colloid science. The structure and behaviour of non-ionic surfactants in apolar hydrocarbons was studied: usually these amphiphilic derivatives (substituted stearates and steroids) give thermoreversible gels once the concentration of aggregates is high enough. These aggregates are constituted by long rigid rods the cross-section of which is function of the solute used. Attention was focussed on the still imprecise but important question of how the solvent nature influences the aggregation process. Small-angle experiments (neutrons and X-rays) have clearly demonstrated the role of steric factors (Fig. 89). On one hand, large structural modifications (radii of gyration and size polydispersity of aggregates) are observed for only one methylene group difference in the solvent structure (cyclohexane), on the other hand, this slight difference is sufficient to induce strikingly different thermodynamic behaviours (mesophases growths and demixions) (ILL).

## Correlations in micellar solutions under shear

Rodlike micelles formed by N-Hexadecyloctyldimethyl-ammonium bromide in aqueous solution were aligned in a shear gradient Γ. The 50 mM solution shows a pronounced peak in scattering intensity having its origin in particle interaction. With increasing shear the intensity distribution becomes very anisotropic, as shown in Fig. 90.

Apart from the correlation peaks seen in this figure a further one at larger scattering angles was observed. An analysis shows that above a threshold shear $\Gamma_0$ a new, highly crystalline phase begins to emerge, being in equilibrium with the phase stable at $\Gamma$=0. The sharp peaks visible in Fig. 90 are characteristic of the crystalline structure, whereas the ringlike feature is due to the $\Gamma$ = 0 phase (Bayreuth).

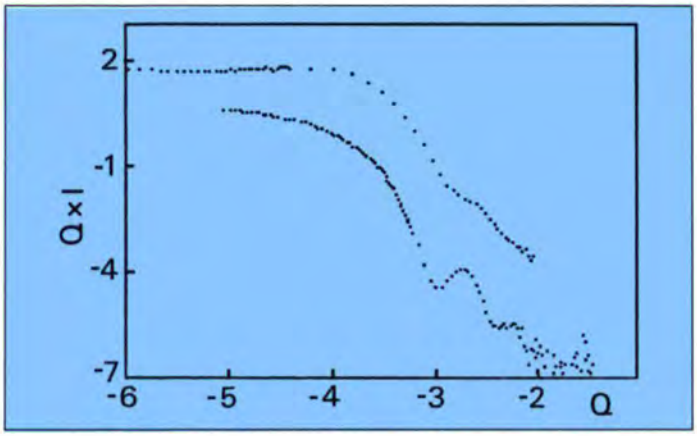


*Figure 89 Specific double logarithmic representation for rod-like aggregates: upper scattering curve, steroid aggregates in cyclohexane (neutrons); lower curve, gel phase in ethylcyclohexane (X-rays).*

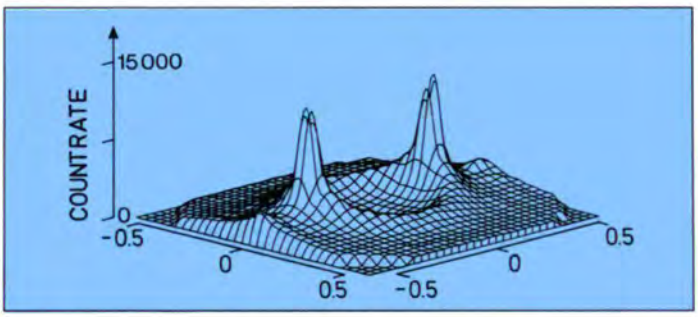


*Figure 90 Isometric plot of the measured scattering intensity of N-Hexadecyloctyldimethylammonium bromide at a shear gradient $\Gamma$ = 400 $s^{-1}$. The scattering vector is given in units of $nm^{-1}$.*

## Structure of fractal aggregates under static and shear flow conditions

Colloidal dispersions composed of aggregates of primary particles are important in a variety of natural and commercial processes. Although it has recently been shown that at high dilution these systems frequently have scattering behaviour which is characteristic of a fractal structure, where the intensity scales with a power law in Q over a range corresponding to the region of self-similarity, the microstructure of concentrated dispersions and its relationship to bulk rheological properties is only poorly understood. SANS has recently been used to probe the structure of concentrated alumina and silica sols under

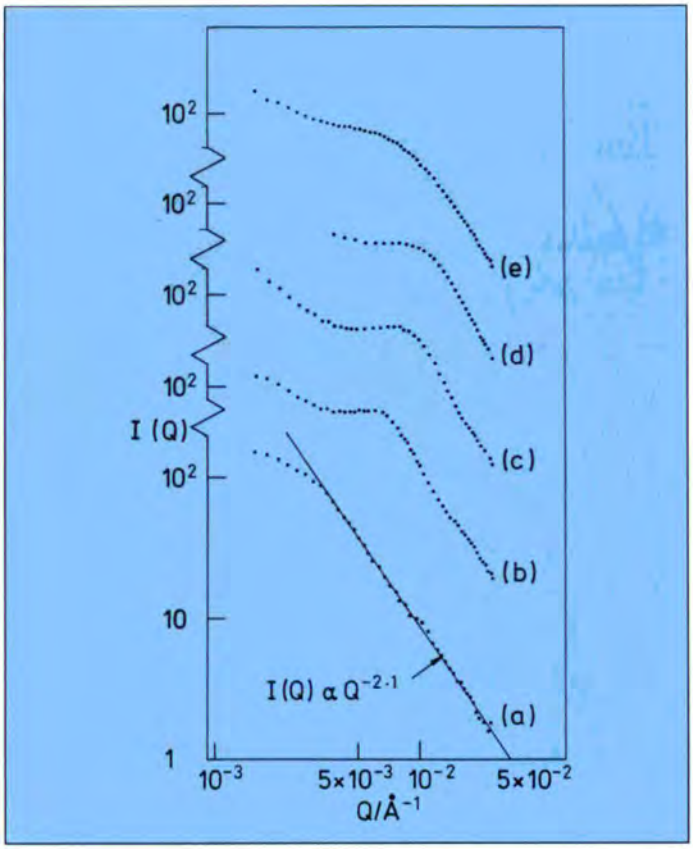


*Figure 91 SANS of aggregated alumina sols of different concentration ($gml^{-1}$). (a) 0.05, (b) 0.2, (c) 0.3, (d) 0.4, (e) 0.4, ionic strength ~ 0.1 mol $dm^{-3}$.*

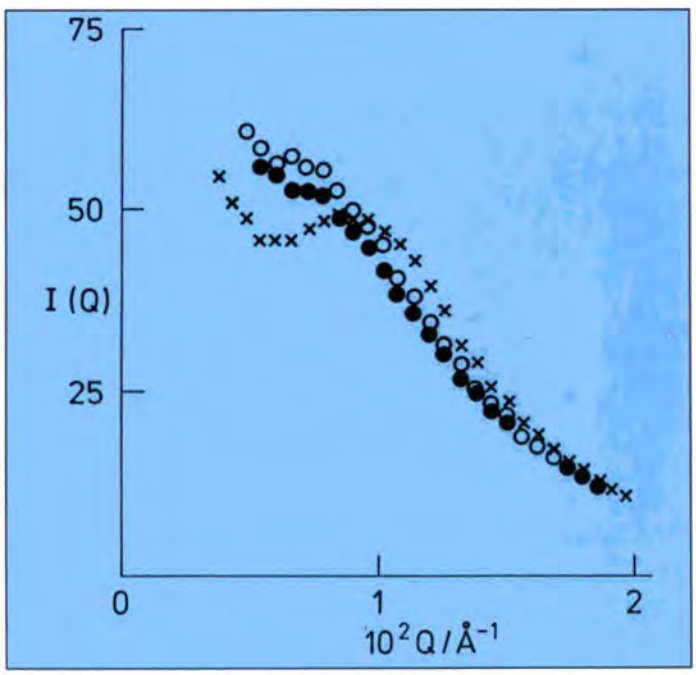


*Figure 92 SANS from concentrated aggregated alumina sol (0.4 $gml^{-1}$); ○ and ●: anisotropic scattering in // and ⊥ orientation to shear gradient $\Gamma$ = 500 $s^{-1}$; ×: under static conditions.*

static and simple shear flow conditions. The rheology of these aggregated sols, which contain primary particles with a diameter of approximately 10 nm, is very dependent on pH and ionic strength, and concentrated dispersions often exhibit shear thinning and thixotropic behaviour. Fig. 91 illustrates the scattering from aggregated alumina sols at different concentrations under static conditions. At the lowest concentration the behaviour is typical of a fractal system, however, as the concentration is increased an interference maximum develops indicating interparticle repulsion and short range ordering between the particles in distinct aggregate units. Such features are dependent on the pH, ionic strength and shear. Under shear the structure is reversibly disrupted as is illustrated in Fig. 92 by the anisotropic scattering from a concentrated sol (0,4g $ml^{-1}$) in a shear gradient $\Gamma$ = 500 $s^{-1}$ and subsequently under static conditions (Harwell, ILL).

## Sheared hard sphere dispersions

Colloidal silica particles sterically stabilized with octadecyl chains grafted to the surface were dispersed in cyclohexane. The particle interactions can be described with the so-called hard sphere model. These types of dispersions are ideally suited to test theories that describe the rheological properties in relation to the dispersion microstructure. In two small-angle neutron scattering experiments on D11 the distortion of the particle structure factor was measured as a function of volume fraction and Peclet number (i.e. shear rate). Since the structure factor becomes anisotropic under shear one has to do measurements with different geometries. Both a Couette set-up and a parallel plate set-up were used. The parallel plates are vertical and the axis of rotation is in a horizontal plane parallel to the neutron beam. By changing the angle between the rotation axis and the neutron beam from -55° to +55° it was possible to map out the structure of a large part of the flow plane. Results show a clear distortion, with an increased density along the compressional axis. Also in the direction perpendicular to the flow plane a slight increase in density is observed. Actually it can loosely be described as a slight temporal clustering. Further, the results show that the problem is highly complex and that ordering phenomena are much less prominent than conjectured in the literature. It also shows that the translation of non-equilibrium molecular dynamics computer simulation results to colloidal systems must be carried out with uttermost care (Utrecht, ILL).

## Influence of molecular mass and crosslinking on the coalescence of latex films

Latex films containing a small amount of perdeuterated latex particles of P-d-nBMA in protonated matrices were examined by small angle neutron scattering. Determining the radius of gyration $R_g$ of the deuterated latex particles as function of temper time and temperature, one can judge upon the interdiffusion of material from different latex particles. Investigation of very weekly crosslinked matrix materials (0.02, 0.04, 0.06, 0.08, 0.1, 0.2, 0.5 weight % methallyl methacrylate, MAMA) showed no change at all neither in the rate of the smearing out process nor in the scattering behaviour as compared to the uncrosslinked sample. In these matrix materials the fraction of very high molecular weight "microgel" increases with increasing MAMA content, as known from ultracentrifuge measurements. From this result it seems that the crosslinked part of the matrix material does not influence the interdiffusion process. Investigation of uncrosslinked matrix materials of molecular weight 33,000 < $M_w$ < 540,000 confirmed the strong $M_w$-dependence of the interdiffusion process found earlier (Ludwigshafen, ILL).

## Polymer melt - chain deformation in shear flow

Melts of polystyrene ($M_w$ = 90000) containing labelled chains were submitted to a steady shear flow (T = 130°C) in a parallel plates geometry. The chain orientation was frozen by rapid quenching. Specimens were cut from the disc at various distances from the centre corresponding to different values of shear rates ($2.5 \times 10^{-3} < \Gamma/s^{-1} < 10^{-2}$). Small-angle neutron scattering measurements were carried out at D11 with the scattering vector Q in the plane of the principal shear directions.

Fig. 93 shows an example of a scattering pattern. It appears that at small scattering vectors Q the main direction of the chain orientation lies close to the shear plane, whereas at higher Q-values the angle between the chain orientation and the shear direction tends to 45°. The interpretation of these experimental results with rheological molecular models is in progress (Strasbourg).

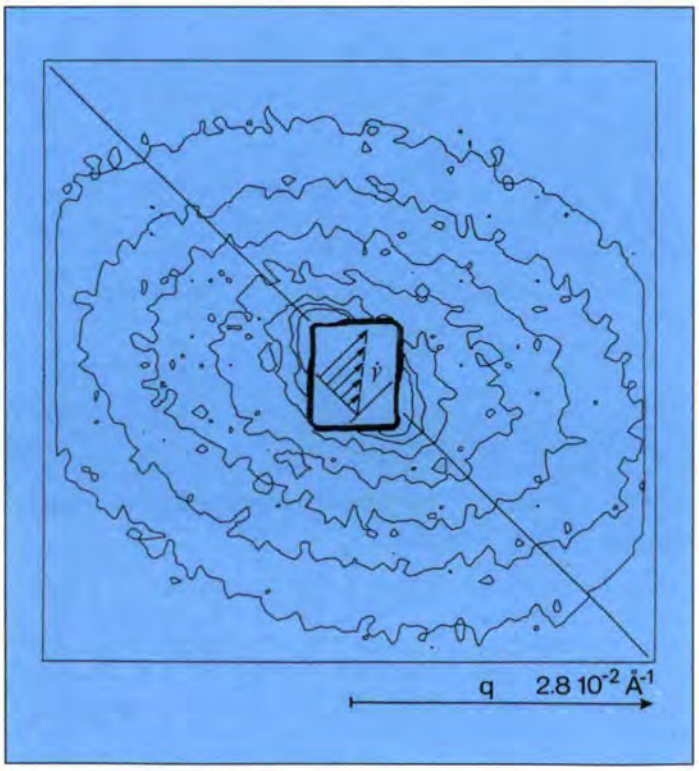


*Figure 93 Scattering pattern of deformed chains in a polymer melt, submitted to steady shear flow and subsequently quenched.*

## Chain conformation of main chain liquid crystalline polyesters

Liquid crystalline polymers consisting of rod-like backbones with flexible side chains are of great scientific interest because their melting point lies below their decomposition temperature and they are soluble in common solvents. Mixtures of protonated and perdeuterated poly (1,4-phenylen-2,5-dialkoxyterephthalate) have been studied in bulk with small-angle neutron scattering at D11 and D17 in order to determine the radius of gyration and the persistence length in the various solid and liquid crystalline phases. Despite of some difficulties due to sample preparation which afflict the small Q-region by aggregation affects, the results at the higher Q-values, however, strongly point to a plate-like shape of the polymer molecules in accordance with deductions from wide-angle X-ray data.

Results obtained from solutions of the protonated material dissolved in deuterated chlorobenzene can be represented by a straight line in a Kratky-plot and hence indicate the high stiffness of the backbone. From these results the average length of the polymers can be estimated to be greater than 400 Å (Mainz, ILL).

## Interaction parameter in polymer blends

The determination of the interaction parameter of polymer blends after Scholte and de Gennes revealed non-linear scattering curves at low Q-values. Similar effects have been observed with scattering experiments on semi-dilute and concentrated polymer solutions. The excess intensity is due to a non-statistical distribution of the centres of gravity of the molecules. For the system d-PMMA/PSAN-19 (perdeuterated polymethylmethacrylate/poly-(styrol-acrylnitril) with 19 weight-% acrylnitril) the correlation length of this long-range concentration fluctuation is of the order of the radius of gyration and about a factor 4 to 12 larger than the correlation length of the single molecule. A scattering function of the form:

$$I(Q) \propto \frac{1}{Q^2+\xi^{-2}} + \frac{k\,a^3}{1 + a^2Q^2}$$

describes the measured scattering curves sufficiently and allows a determination of the structure factor of the single molecules and of the interaction parameters. In the transition regime with 40% d-PMMA the shape of the scattering curve is strongly dependent on the sample preparation. Only the non-linear scattering curve can be attributed to the thermodynamically stable state (Fig. 94). No excess intensity is observed if the PMMA is only *partly* labelled with deuterium, in analogy to experiments with polymer solutions (Mainz).

## Concentration fluctuations in compatible polymer mixtures

The excess structure factor of a binary polymer mixture consisting of polymethylstyrene and polystyrene has been measured by small-angle neutron scattering. In accordance with a mean-field treatment it has been found that the concentration dependence of the excess structure factor can be very well described by the Flory Huggins equation. But looking at Fig. 95a it is obvious that a simple $\Phi$ $(1-\Phi)$-dependence would fit the data as well. Due to the lack of a theoretical interpretation of this mathematical form it has been decided to use the Flory Huggins equation as an adequate way of interpreting the results. As attention has been focussed on the molecular weight dependence (Fig. 95b), it seems that this theoretical treatment is not perfectly capable of describing the data. The molecular weight dependence of the $\chi$-parameter (Fig. 95c) shows that this discrepancy is mostly prominent in the smallest molecular weight sample. A good agreement with the Flory Huggins theory is found for higher molecular weight compounds (Mainz, ILL).

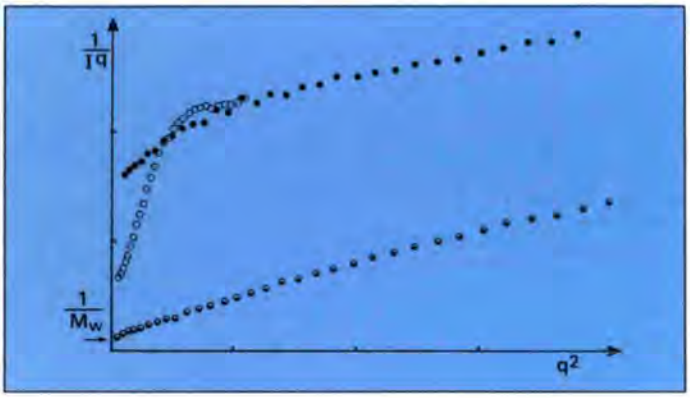


*Figure 94 Reciprocal scattering intensity (arbitrary units) as a function of $Q^2$: 40% d - PMMA before (○) and after (●) annealing, 34% h - PMMA and 1 % d - PMMA (◒).*

## Internal chain relaxation in PDMS-melts

One of the major problems still unsolved in the dynamic of polymers concerns the nature of the molecular motion in dense systems. Intuitively it is clear that topological constraints mutually imposed on each other by the different moving chains must play a decisive role in determining the motional characteristics of the chains.

The most popular model in this area is the reptation model which describes the topological hindrance in terms of a tube around the profile of a given chain. Chain motion is then only possible inside a spatially fixed tube the lifetime of which may easily reach macroscopic times. While viscoelastic theory based on such a reptation concept was very successful to describe rheological properties, microscopically the situation is much less clear. Theoretically one expects a crossover from Rouse relaxation at times shorter than that a chain segment needs to equilibrate across the tube diameter, to a so-called local reptation regime where density fluctuation equilibrate along the fixed tube. The most important feature of this microscopic reptation concept is the introduction of a new microscopic length scale in between the chain dimension and the elementary length of a polymer segment.

Up to now microscopic dynamic studies of chain relaxation revealed the basic features of Rouse relaxation (characteristic

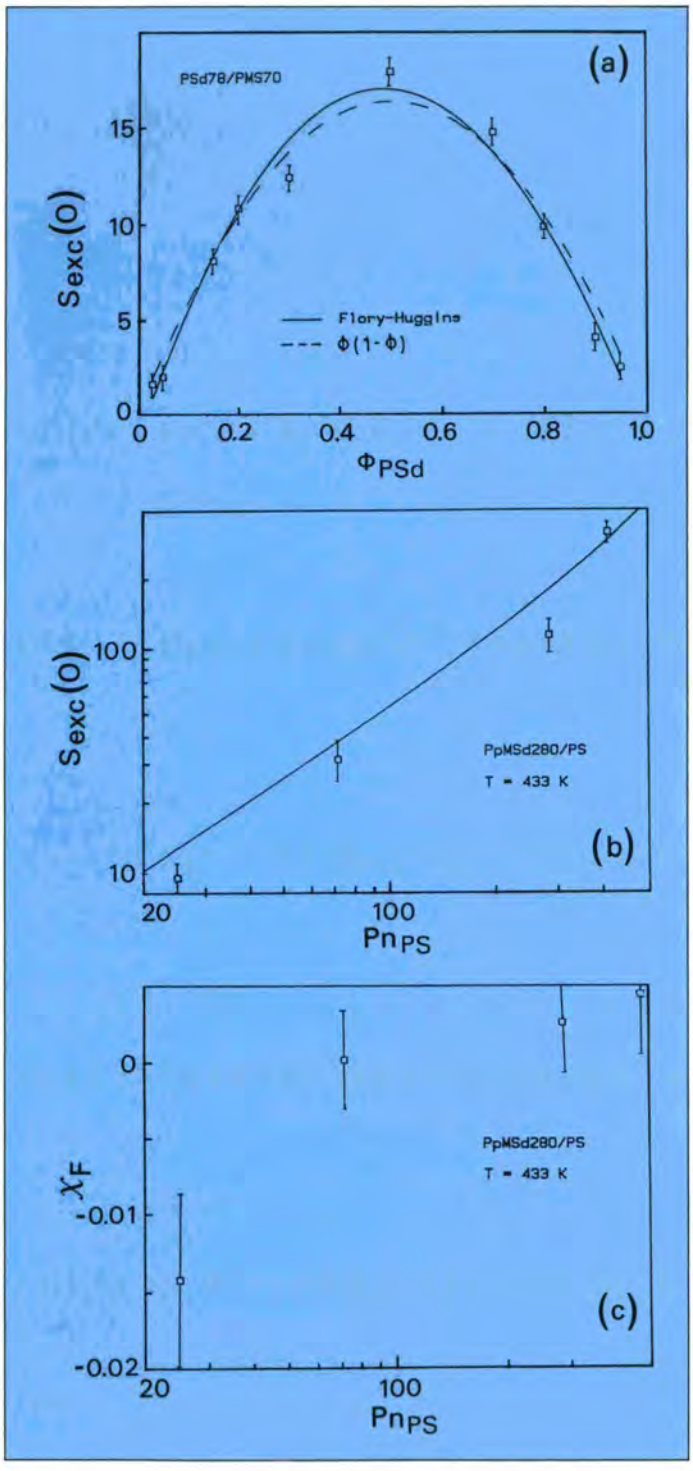


*Figure 95* *a) Concentration dependence of the excess structure factor from a binary mixture of deuterated polystyrene (degree of polymerization = 78) and polymethylstyrene (degree of polymerization = 70) at a temperature T = 433 K.*
*b) molecular weight dependence of the excess structure factor from a binary mixture of deuterated poly-para-methylstyrene (degree of polymerization = 280) and polystyrene (different degrees of polymerization) at a temperature T = 433 K.*
*c) interaction parameter χ as a function of the degree of polymerization of polystyrene.*

frequency $\Omega_R \sim Q^4$) although deviation in the spectral line shape appeared to be evident. Increasing the resolution of IN11 and accelerating the mobility in PDMS by heating to 200°C the internal chain relaxation in PDMS-melts was reinvestigated on a timescale which was larger by one order of magnitude. The investigated deuterated melt had a molecular weight of $M_w$ = 160, 000 with $M_w/M_n$ = 1.2. The added protonated fraction (5%) is characterised by $M_w$ = 150, 000 and $M_w/M_n$ = 1.2. Fig. 96 displays the experimental results in a scaling representation.

For Rouse relaxation all experimental data should scale with

$$u = Q^2 \sqrt{W\sigma^4 t},$$

where W is the Rouse rate and σ is the segment length. The solid line presents the relaxation function of the Rouse model. This function has been fitted in the region of $u < 5$ where the data follow the Rouse prediction $\Omega \sim Q^4$. As can be seen in this low u region the data are very well represented by the Rouse function while at higher u systematic deviations towards slower relaxation appear. One could take this as a signature of topological constraints which prevent fast relaxation at high u. On the other hand one realizes that even at high u the data obey the

$$u = Q^2 \sqrt{W\sigma^4 t} \quad \text{scaling.}$$

In this respect the experiment is at variance with the fundamental prediction of local reptation, namely the existence of an additional length scale. Such a second microscopic length

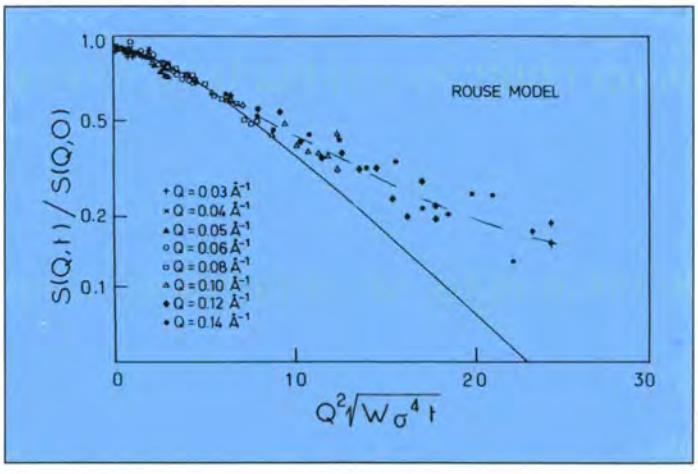


*Figure 96* *Scaling representation of the intermediate scattering function from a PDMS melt as obtained on IN11. The solid line presents the prediction of the Rouse model. The dashed line serves as a guide to the eyes.*

scale would result in a splitting of the relaxation curves with different Q in the scaling plot. Thus, although the data indicate strong influence of topological constraints, they do not show evidence for a further length scale represented by the tube diameter (ILL, Mainz).

## Polymer diffusion in porous media

Diffusion of polymer chains inside porous materials control many practical problems such as permeation or filtration, but very little is known about the conformation of the chain when the pores are very small as well as about the dynamics of the chain segments in such a restricted geometry. An experimental investigation of these questions started at Jülich and at the ILL, using SANS and Spin-Echo Spectroscopy on solutions of Polybutadiene inside Aerogels.

The solvent is a mixture of benzene and deuterated benzene in proportions calculated to match the aerogel. From desorption analyses, the aerogel is estimated to have an average pore size of the order of 80 Å.

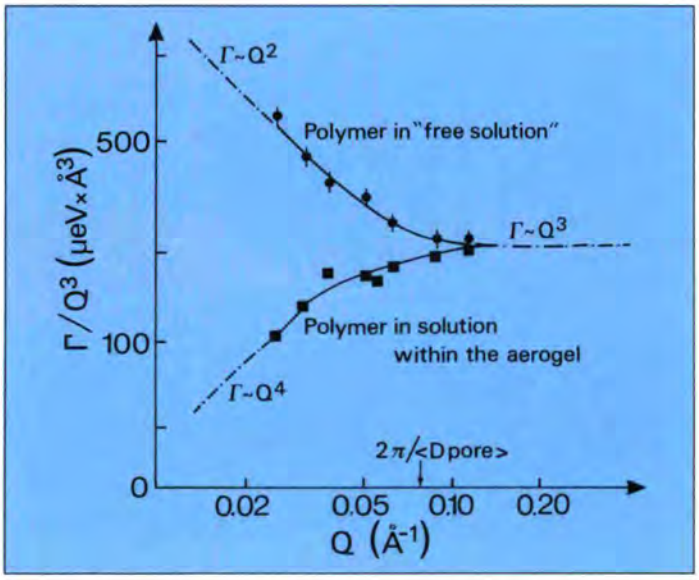


*Figure 97 Double logarithmic plot of the reduced relaxation frequency $\Gamma/Q^3$ versus momentum transfer Q, from NSE measurements at $\lambda$ = 8.6 Å and T = 330 K, after subtraction of the measured background and fit of the echo amplitudes with a Zimm model: $(S(Q,t)/S(Q,0) = \exp(-[\Gamma t]^{0.7})$. The solution contained 5% in weight of Polybutadiene with molecular weight 50,000.*

Fig. 97 summarizes the results of Neutron Spin-Echo measurements of the polymer dynamics inside the aerogel, compared to those of the polymer dynamics in the "free solution".

While, at large Q, both results converge asymptotically towards Zimm behaviour ($\Gamma \sim Q^3$); at small Q, the chains in "free solution" exhibit a collective relaxation spectrum ($\Gamma \sim DQ^2$), whereas the chains in the aerogel appear to reveal only decoupled entropy controlled (Rouse) modes ($\Gamma \sim Q^4$). Some ambiguity in the interpretation however remains, due to the fact that in both systems the apparent cross-over regimes take place in the same Q range. Therefore, further investigation will concentrate on polymers with different molecular weight inside aerogels with different pore sizes (Jülich, ILL).

## Conformationally disordered phases in crystallized fluoropolymers

Linear chains of fluorinated copolymers like: $-(CH_2\text{-}CF_2)_m\text{-}(CHF\text{-}CF_2)_n-$ or $-(CH_2\text{-}CF_2)_m\text{-}(CF_2\text{-}CF_2)_n-$ generally crystallize into well-ordered structures, but also exhibit intermediate plastic-like phases below their melting point. In these conformationally disordered crystalline structures a high translational order is conserved in the hexagonal plane perpendicular to the chain axis, but fast reorientation of the chain segments almost destroys the translational symmetry along the chain axis. These phases therefore represent a kind of model system to investigate the local dynamics in amorphous polymers. Their density is comparable to that of the amorphous phase and NMR investigations have shown that the reorientation of the chain segments takes place in the same frequency range as in the amorphous phase (Strasbourg and Montana St. Univ.). When cooling the copolymers with a high $CH_2\text{-}CF_2$ content, a strong first order transition is observed towards an ordered phase (with orientational domains); but when cooling copolymers with a low $CH_2\text{-}CF_2$ content, a continuous glass-like transition is observed towards a crystalline phase with apparently no orientational order (Bell Labs.).

In the conformationally disordered crystalline phases, long-range translational diffusion can be neglected. It is therefore

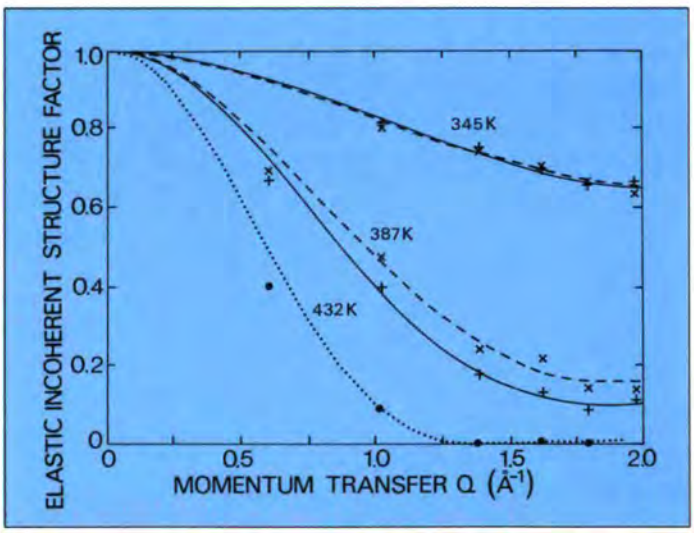


*Figure 98 Experimental EISF at different temperatures for two orientations of a drawn P(VF2-F3E) copolymer with composition 70/30 mol.%, and results of a fit with a model of diffusion inside a cylindrical volume of radius R and height H:*
*(+) and continuous lines correspond to the chain axis perpendicular to the scattering plane,*
*(×) and broken lines correspond to the chain axis in the scattering plane,*

*Results provided by the fit:*
- *disordered crystal phase at 387 K,*
  *R = 2.0 Å, H = 3.3 Å, 13% of fixed protons,*
- *ordered crystal phase at 345 K,*
  *R = 1.5 Å, H = 2.5 Å, 63% of fixed protons,*
- *molten phase at 432 K,*
  *R ≈ 2.7 Å, 0% of fixed protons.*

possible to analyze the incoherent quasi-elastic neutron scattering using models of diffusion of the protons on a restricted number of sites. Neutron studies have started on the copolymer, with composition $(CH_2\text{-}CF_2)_{0.7}\text{-}(CHF\text{-}CF_2)_{0.3}$ in which the disordered phase extends from 420 K to 350 K.

After a first investigation of the overall dynamics of powder samples using the instruments IN6 et IN10 (Grenoble, MSU and ILL), a more detailed study was recently performed on IN10 using oriented specimens with fibre symmetry (Grenoble, Strasbourg and ILL). Fig. 98 shows the Elastic Incoherent Structure Factor in the planes perpendicular and parallel to the chain axis, fitted with a model of diffusion inside a cylindrical volume. These results show that in the disordered phase (387 K) the protons experience motions of almost comparable amplitudes along the chain axis and around the chain axis. When the crystalline phase is ordered (345 K), a small and isotropic decrease of the EISF is however observed. It is attributed to motions in the amorphous phase anchored at the surface of the lamellar crystals. In the molten phase (432 K), in principle an EISF cannot be extracted, but the results obtained with the same treatment are shown to evidence the further change in the motions of the chains.

These investigations are continuing in different directions:

(i) measurements of the IQNS on copolymers with different compositions to analyze the changes in the dynamics (Madrid and ILL),

(ii) selective deuteriation of the monomers to analyze the motion of just one type of protons and the quasi-elastic coherent scattering (MSU, Bell Labs and ILL),

(iii) effects of a hydrostatic pressure on the characteristics of the order-disorder transition (Grenoble, Strasbourg and ILL).

Secretary: P. Lindner

## On the Dynamics of Microemulsions

B. Farago, D. Richter

The existence of microemulsions is far from being a recent discovery, but they are still in the focus of interest of physics. The recipe is very simple; put together water and oil and you have a two phase liquid, add some surfactant, shake it, and if you have chosen your components well you might end up with a single phase water clear liquid. In a first approximation the thing is very simple. The surfactants are molecules, in general, with a hydrophylic head and a hydrophobic tail. The head prefers to be in water as the tail does its maximum to stay in the oil so they arrange themselves to be in the water-oil interface and if the amount of surfactant is correctly chosen one can solubilize the oil in water or inversely. For example cf. the volume of the oil is much less than that of the water, the most likely structure is that small oil droplets are surrounded by the surfactant molecules and if the size of the droplets is much smaller than the wavelength of the light they become transparent [1].

Of course the real nature is more complicated than that. First of all the recipe does not work all the time. Sometimes one has to add so-called cosurfactants (eg. short chain alcohol) to arrive at microemulsion formation, or the resultant liquid spontaneously separates in two phases, both of them being a homogeneous mixture of oil-water-surfactant. The rheological properties can be very different too, from liquid like viscosity up to gel like behavior.

The first efforts went in the direction of characterization of the structures. Here the small-angle neutron scattering proved to be an essential tool. First of all because the characteristic length scale of these systems is around 100 Å, secondly because with selective deuteration (oil-water-surfactant) one can get distinct information about the structure of the different composants. It turned out that depending on the composants and concentrations a great variety of structures exist: spheres as mentioned above, but also tubes, lamellar structures or more complicated interpenetrating bicontinous structures. Taking into account that the microemulsions might contain as much as 100 $m^2$ surface in 1 $cm^3$ it is clear that the proprieties of this surface must be determinant in the physics of the microemulsion formation.

With the advent of the high resolution Neutron-Spin-Echo spectroscopy (NSE) besides the structural studies the dynamics also became accessible for neutron scattering.

In fact, on one hand, one can argue that the surface tension of the oil-surfactant-water surface must be extremely low otherwise thermodynamics would forbid the existence of such big surfaces [7], on the other hand the structural studies have shown that we are dealing with saturated surfaces, that is to say that practically all the surfactant molecules are in the water-oil interface and the surface per surfactant molecule is a fixed quantity [3]. In this sense one could speak of a static surface tension (energy = surface x surface tension) which should be low, and dynamical surface tension which could be measured by deforming the droplets from their equilibrium

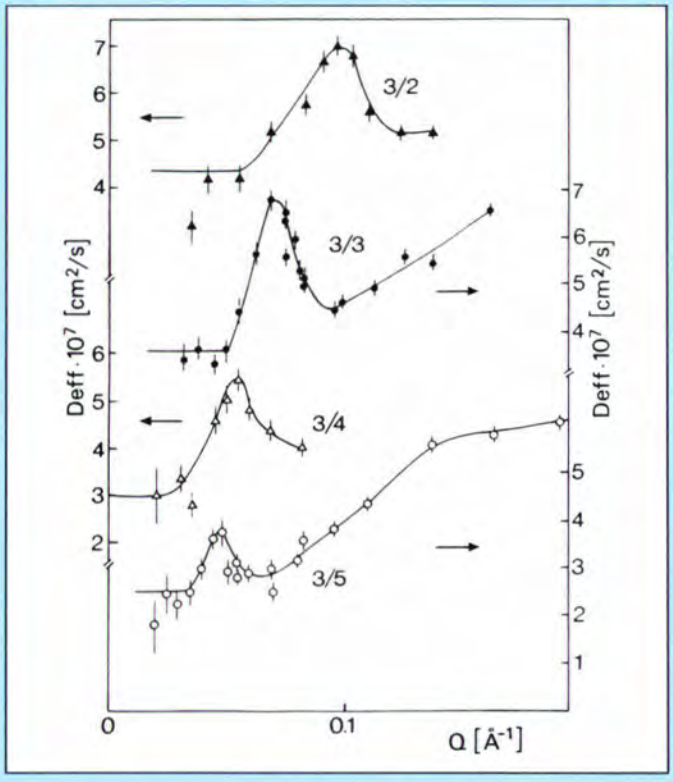


*Figure 99 The measured effective diffusion coefficient as a function of q for the different shell sizes.*
▲ $r_0 = 34.4$ Å
● $r_0 = 48.7$ Å
Δ $r_0 = 59.2$ Å
○ $r_0 = 70$ Å

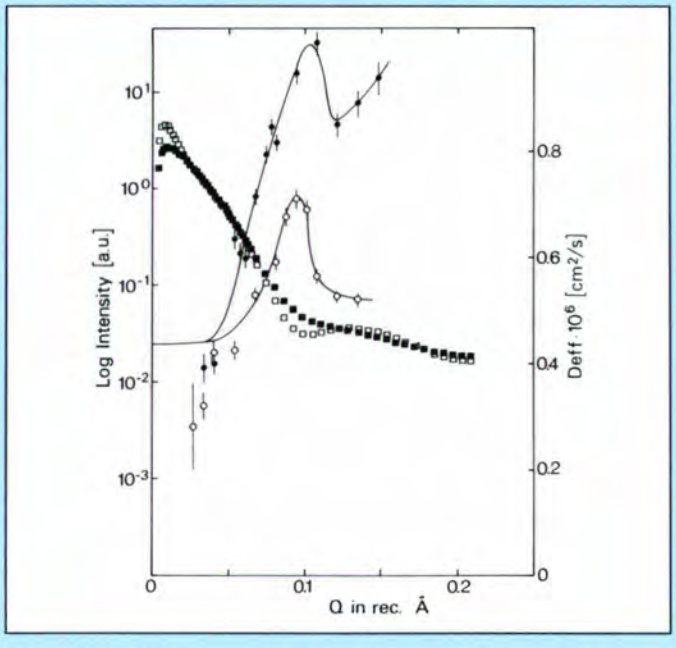


*Figure 100 SANS spectra and $D_{eff}$ versus q for the $r_0 = 34.4$ Å shell (□ and ○) and the $r_0 = 32$ Å buthanol diluted shell (■ and ●) samples. Lines are guides to the eye.*

shape. In fact, if the surface area per surfactant molecule is solely determined by its chemical structure, by deforming the droplets we bring the water directly in contact with the oil and this dynamical surface tension can be of the order of the water-oil surface tension which is high.

Recent theoretical work achieved considerable progress in describing the phase diagram of microemulsions where the deterministic parameter is the so-called bending energy which is associated with the energy needed to deform the surface from its preferred curvature [4]. This latter is a function of the actual surfactant molecule (e.g. head size compared to the tail volume).

A decision between the ideas of surface tension or bending energy controlled microemulsion formation can be obtained experimentally if the thermal shape fluctuations are studied. From such experiments one may learn which one is the determining driving force.

Let us consider microemulsions which on time average have a spherical shape. The scattering form factor of a sphere is given by $(3j_1(qr)/qr)^2$, where $j_1(x)$ is the first spherical Bessel function, q the momentum transfer and r is the droplet radius. This function has its first zero value at $qr = 4.48$. If the sphere exhibits thermal fluctuation than they should be visible best in this q-range because there the scattering from the droplet structure has its minimum. We can reach a further enhancement if only the surfactant shell is labelled (protonated surfactant with deuterated oil and water), then the neutrons only see the surfactant shell. In this case the scattering formfactor is $j_0(qr)$ which has its minimum at smaller q ($qr = \pi$) and we may observe the lowest order excitations. Studying at both contrasts we can verify that the observed inelasticity is really due to shape fluctuations. Again the reality is more complicated since the zero of the form factors is reduced to a shallow minimum due to the polydispersity of the samples. Nevertheless our arguments remain valid and we should look for shape fluctuations in these qr regions.

The shape of the droplets always can be described in terms of spherical harmonics:

$$r(\theta,\phi) = r_0 + a_{lm} Y_{lm}(\theta,\phi)$$

Assuming small amplitudes we can write the intermediate scattering function, which is measured by NSE as:

$$I(q,t) = \exp(-Dq^2t)\, V^2 \rho^2 [f_0(qr) + \Sigma(2l+1)/4\pi r^2 f_l(qr) \langle a_l(0)a_l(t)\rangle]$$

Where:

$f_0(qr) = [3j_1(qr)/qr)]^2$ $\qquad$ $f_l(qr) = [3j_l(qr)]^2$

for the full sphere, and

$f_0(qr) = [j_0(qr)]^2$ $\qquad$ $f_l(qr) = [(l+2)j_l(qr)\text{-}qr\, j_{l+1}(qr)]^2$

for the shell contrast.

All the $f_l(qr)$ ($l > 0$) approach zero as q goes to zero. Thus at small q the time averaged $f_0(qr)$ is dominant and the intermediate scattering function can be described by a single exponential decay $\exp(-\Gamma t)$ with $\Gamma = Dq^2$ where D is the translational diffusion coefficient. At higher q where the other terms become important, we expect an enhancement of the effective diffusion coefficient $D_{eff} = \Gamma/q^2$. As can be seen from Fig. 99 and 100 indeed we find a peak in $D_{eff}$ in the region of the minimum of the static form factor. The position of the peak scales with the size of the shells and for a given radius it shows up at different q for the shell and for the sphere sample.

Without going in the details of the calculation of the $\langle a_l(0)a_l(t)\rangle$ amplitude, with a simple dimensional analysis we can make a guess what can be expected for surface tension driven and for bending energy driven fluctuations.

The characteristic relaxation rate should scale as

$$\tau^{-1} \sim K/\eta r_0^3 \qquad\qquad \tau^{-1} \sim \sigma/\eta r_o$$

where $\eta$ is the viscosity, K is the curvature elastic modulus, and $\sigma$ is the surface tension. The line width due to the translational diffusion at $qr_o = \pi$, $Dq^2$ , scales as $r_0^{-3}$ so the ratio of peak height to diffusion is constant if the bending energy is the driving force and should scale with $r_0^2$ for surface tension driven fluctuations.

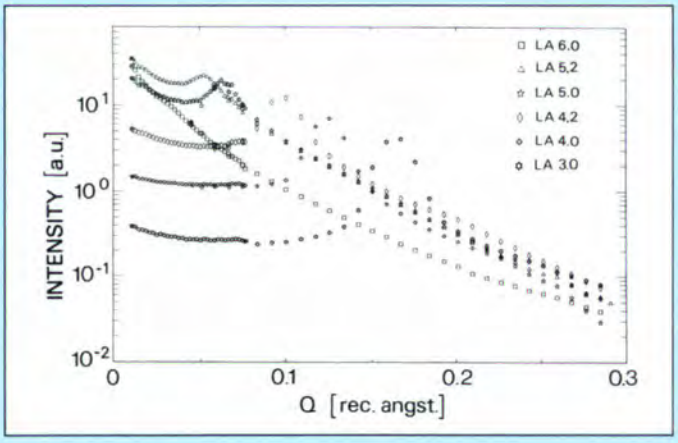


*Figure 101 SANS spectra of the lamellar micro-emulsions; d is the interlamellar distance.*
*LA3 d=37Å*
*LA4 d=50Å*
*LA4.2 d=63Å*
*LA5 d=98Å*
*LA5.2 d=126Å*
*In the case of the LA6 sample the first neighbour peak has completely disappeared, hence this sample was used to test the excitation spectra of the free film.*

The model system we have chosen is the classical decane-AOT-water water in oil microemulsion. We varied the mean radius from 16 Å to 70 Å and as it can be seen from Fig. 86 the peak to diffusion ratio for all of them was around 1.6. This result proves the dominating role of surface elasticity in the formation of microemulsions [5]. Detailed calculations give a value for $K \sim 9kT$.

The next question we ask is, what is the role of the cosurfactants? To answer this question we added buthanol (1:1 AOT:buthanol) to the $r_0 = 48.7$ Å shell sample.

Using small-angle neutron scattering one can immediately see that:

a) the average size is reduced, in fact comparing with the $r_0 = 34.4$ Å sample, the minimum is almost exactly at the same position.

b) the minimum is much less pronounced which tells us that the polydispersity is higher. With the same bending energy K a higher polydispersity would smear out the peak structure found for $D_{eff}$ as a function of q. In fact what we see is quite the opposite (see Fig. 100) the peak is very much enhanced. On the other hand the smaller radius indicates that the apparent surface area per AOT molecule is increased. The common explanation is that the alcohol molecules are situated between the surfactants making more surface available and reducing at the same time the surface rigidity to about 1 kT. This is in agreement with theoretical calculations which predict an increasing polydispersity with decreasing rigidity. In view of this rsult we think that a cosurfactant is needed for microemulsion formation cf. the surfactant alone forms too rigid a layer.

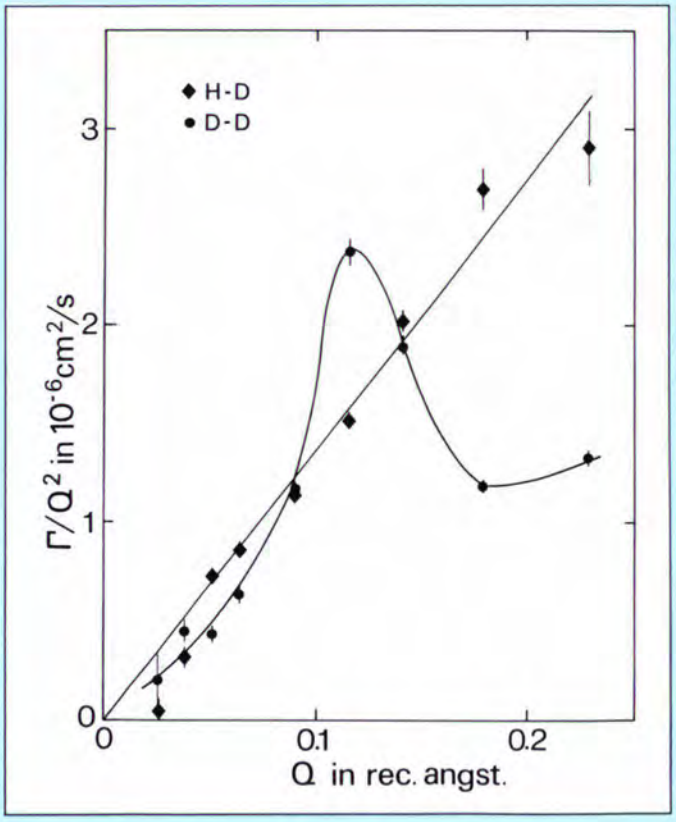


*Figure 102 $\Gamma/q^2$ measured by NSE on the LA6 sample with*
- ◆ *$H_2O$ - SDS - deuterated dodecane /full film/*
- ● *$D_2O$ - SDS - deuterated dodecane /double layer film.*

For lamellar structures the theory of overdamped capillary waves predicts a $q^3$ dispersion relation [6]. An experimental example for a lamellar system is the water-SDS-dodecane-penthanol system. Here the water with the surfactant forms a double layer embedded in an oil matrix. By changing the oil content one can change the interlamellar distance as can be seen from the displacement of the correlation peak in the SANS spectra (Fig. 101) (deuterated water-protonated SDS and dodecane). Inelastic experiments on a compound built of $H_2O$, protonated SDS and deuterated dodecane lead to a nearly perfect $\Gamma \sim q^3$ dispersion relation (Fig. 89). The surprise came when we repeated the experiment this time with $D_2O$-protonated SDS-deuterated dodecane, that is to say the neutrons see the membrane as a double layer. We have found again a strong peak in $D_{eff}$ at the q position which corresponds to the breathing mode of the double layer (Fig. 102) the existence of which was not expected.

This example shows that in spite of the considerable progress we are still far from the understanding of these fascinating objects. Further experiments in particular an ordered lamellar or rod like structures are expected to give deeper insight into the intra and inter lamellar or rod forces. Thereby NSE will maintain its unique role. It is hoped that these experiments will also encourage the theoretical work needed to comprehend these structures.

## References:


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# Instrument Operation Department

# Instrument Operation Department

## Einleitung

Die Abteilung Instrumentenbetrieb hat im wesentlichen drei Aufgaben :

1. Bereitstellung von Geräten zur Probenumgebung.

2. Technische Unterstützung der Wissenschaftler bei ihren Experimenten.

3. Instandhaltung und Ausbau des Geländes, der Gebäude und der technischen Einrichtungen (mit Ausnahme des Reaktors).

Auf dem Gebiet der Probenumgebung hält weiterhin die Nachfrage der Wissenschaftler nach immer schwieriger zu erfüllenden Bedingungen an. Um dieser Nachfrage gerecht werden zu können, sind zusätzliche Investitionen von Sach- und Personalmitteln nötig. Letztere werden dem ILL indirekt durch die Unterstützung durch externe Laboratorien geliefert (Beispiele : Walther-Meissner-Institut für Tieftemperaturforschung in Garching ; CRTBT in Grenoble).

Die technische Unterstützung der Wissenschaftler bei der Durchführung ihrer Experimente wird von unseren Instrumenttechnikern gewährleistet. Unter der Leitung von ILL Wissenschaftlern sind sie für den Betrieb, die Entwicklung und die Instandhaltung der Instrumente verantwortlich. Deshalb spielen die Instrumenttechniker eine sehr wichtige Rolle im Betrieb des ILL. Jede Ansrengung muss gemacht werden, dieses Team motiviert, stark und wohl organisiert zu halten. Im Hinblick auf die Tatsache, dass die Anzahl der ILL Instrumente auf Grund des "Deuxième souffle" (zweite kalte Quelle, zweite Neutronenleiterhalle, etc.) anwächst und im Hinblick auf den Plan, die Anzahl der Stellen für Experimentalphysiker zu erhöhen, ist es klar, dass die technische Unterstützung der Instrumente auch verstärkt werden muss, das heisst : Neue Instrumenttechnikerstellen werden benötigt.

Die Abteilung "Aménagement et Entretien" (SAE) war auf Grund des Baubeginns des ESRF starkem Arbeitsdruck ausgesetzt. Besprechungen mit dem ESRF finden wöchentlich zur Koordination der Aktionen statt, die das gemeinsame ILL/ESRF Gelände betreffen. Trotz zahlreicher Probleme war die Abteilung SAE in der Lage, das für das ESRF vorgesehene Gelände Ende 1988 freizugeben.

## Introduction

Le Département d'Exploitation des Instruments regroupe trois activités essentielles :

1. Fourniture de matériel et d'assistance technique pour l'environnement des échantillons pendant les expériences.

2. Support technique sur les instruments pour les chercheurs en cours d'expérience.

3. Entretien et amélioration du site, des bâtiments et des installations techniques (sauf le réacteur).

Dans le domaine de l'environnement d'échantillons, les chercheurs continuent à demander du matériel plus sophistiqué. Pour satisfaire cette demande, un investissement financier et un investissement en main d'œuvre sont indispensables. Du personnel supplémentaire est fourni indirectement à l'ILL grâce à l'aide de laboratoires externes (par exemple : Walther - Meissner - Institut für Tieftemperaturforschung à Garching, CRTBT à Grenoble).

Le support technique sur les instruments en service est assuré par les techniciens d'instrument. Sous la direction des chercheurs de l'ILL, ils ont la responsabilité du fonctionnement, du développement et de l'entretien de leur(s) instrument(s). Ils apportent assistance au personnel scientifique et aux visiteurs pendant leurs expériences. Par conséquent, ces techniciens ont un rôle très important dans le fonctionnement de l'ILL. Des efforts substantiels sont nécessaires pour maintenir cette équipe motivée, solide et bien organisée.

Etant donné le nombre croissant d'instruments à l'ILL, dû au "Deuxième souffle "(deuxième source froide, deuxième hall de guide, etc.) et dans la perspective d'accroissement du nombre d'expérimentateurs, il est clair qu'il faudra augmenter aussi les effectifs des techniciens d'instrument.

Le troisième secteur d'activité d'EDEX, le Service "Aménagement et Entretien" a été particulièrement sollicité cette année du fait du démarrage des travaux d'installation de l'ESRF à proximité immédiate de l'ILL. Des réunions techniques hebdomadaires sont tenues pour coordonner toutes les actions concernant le site et les bâtiments. Malgré de nombreux problèmes extérieurs, nous avons pu respecter les délais prévus et le site pour l'ESRF a été mis à disposition fin 1988.

## Introduction

The Instrument Operation Department EDEX has three essential activities:

1. Provision of sample environments for the experiments.

2. Technical support on the instruments for the scientists performing their experiments.

3. Maintenance and improvement of site, buildings and technical installations (except reactor).

In the field of sample environment, the demand of the scientists for more sophisticated equipment continues. In order to meet these demands financial and manpower investment is mandatory. Additional manpower is provided to the ILL indirectly via the help of external laboratories (Examples: Walther-Meissner-Institut für Tieftemperaturforschung in Garching, CRBT in Grenoble).

The technical support on the running instruments is provided by our instrument technicians. They are responsible, under the supervision of ILL scientists, for the operation, development and maintenance of their instruments. They assist the scientific staff and visitors with their experiments. Therefore the instrument technicians play a very important role in the operation of the ILL. Every effort has to be made to keep this team motivated, strong and well organized. In view of the increasing number of instruments at the ILL as a result of the 'deuxième souffle' (second cold source, second guide hall, etc.) and in view of the plan to increase the number of experimental scientists at the ILL, it is clear that the manpower for technical support on the instruments must also be increased.

The third sector of activity of EDEX, the 'Service Aménagement et Entretien', has been under very strong pressure this year because of the start of the installation of the ESRF on the site very close to the ILL. Weekly technical meetings take place to coordinate all the actions concerning site and buildings. Despite numerous external problems we were able to respect the time schedule, and the site for the ESRF was liberated at the end of 1988.

# Advanced Cryogenic Service

## Thermometry and Instrumentation

The ILL precision temperature controller (PTC) is now commercially available outside the Institute after the signing of a licence agreement with the manufacturer. Minor modifications are constantly being made to both the hardware and software of the PTC in an effort to satisfy the various requests made by users. It is this constructive feedback which has enabled us to render the controller both easier to use and more reliable.

The testing of a second PID algorithm controlling the sample temperature has been successful and has been installed on many instrument control computers.

## Superconducting Magnets

The 6 Tesla Oxford Instruments cryomagnet was in use for 130 days and has worked well, except for a small leak which occured on the sample tail. This leak was repaired and the tail modified, further modification may be required if problems persist.

The 5 Tesla Oxford Instruments horizontal field cryomagnet has been repaired, the cold leak which existed was eventually found to be on the magnet part of the assembly.

The dilution insert for the 6 Tesla Oxford Instruments cryomagnet is now being tested, a dedicated gas handling system for this insert has been constructed, the complete system should be operational early in 1989.

## Very Low Temperatures

In routine operation there were more than 100 days of measurement at very low temperature (20 mK < T < 1 K) with 30 samples. The rapid sample changing facilities during an experiment are much appreciated.

Construction and development: the construction of the gas-handling system for the high power dilution cryostat (Godfrin project) is in progress. The study for the cryostat proper is in its initial phase; a dilution insert with condensation at 4 K (instead of 1.5 K) has been built and fully tested. Its operation is stable, and the temperature obtained is 20 mK. The system has the disadvantage of requiring a non-standard gas-handling configuration. It will therefore be standardized for condensation at 1.5 K, which will make it interchangeable with the other two inserts.

The 4-circle cryostat for D10 is now in routine operation. Construction has started on a second similar cryostat. This will be able to operate above ambient temperature up to 300°C. Thus a single unit will cover the range 1.7 K to 573 K.

*The second guide hall is being fitted out. In the picture (status October 1988) one can see the guide N° H53 feeding IN10C and the $n\bar{n}$ experiment. In the foreground is the foundation for the IN15 tanzboden.*

# Central Service

## General Intervention Group

### Principal tasks

General organisation and maintenance of the experimental halls in an operational state.

Organisation of all manipulations in the halls requiring cranes and lifting equipment.

Maintenance of the halls: cleaning and painting work.

Management and distribution of gases and cryogenic liquids.

Management and distribution of shielding materials.

Organisation of 'heavy' transport by lorry on the site, and snow clearing work.

Coordination of work in the halls and on the instruments. Preparation and follow-up of the general planning of work and associated constraints.

Management of the ancillary laboratories: low activity lab, X-ray lab, Nuclear Physics group evaporator.

In 1988 a special effort was made, particularly in the following areas:

Dismantling and re-installation of the instruments PN1 and D15, to allow the changing of the H9 beam-tube thimble.

Assembly of shielding associated with the Horizontal Cold Source.

Assistance with the assembly of the instruments IN14, IN10C, nn̄.

Assistance with the equipment in the new guide hall ILL22.

Measurement and reduction of accoustic noise on level C, and the installation of accoustic insulation.

Improvement of the neutron and gamma background noise levels by additional shielding on Gams4, PN3, D9B, D2B, IN1B, H53, IN10C and IN4.

In 1988 the mechanical assistance team, consisting of three members of the general intervention group, gave rapid assistance in many instances. Its action, in the fields of mechanical engineering and assembly of instruments, greatly facilitated the construction or modification work on the instruments, for instance on H21 beam equipment, systematic assistance to the 'S' instruments, improvements on IN4, D4B, D19, DB21, etc.

## Specific Intervention Group

### Principal tasks

Surveillance of the experimental areas, the integrity of shielding and the safe use of beams and samples.

Systematic tests and verification of safety devices on the instruments and of the correct distribution of neutrons before each reactor start-up.

Assistance with the carrying out of high risk experiments that required a tightly controlled environment.

Management and supervision of the parameters and alarms monitored by the reactor control room using the SADI network: preparation of operation and safety instructions.

Measurement of gamma and neutron background levels with a view to improving the biological shielding around the instruments.

In 1988 the following work was carried out:

Modification and extension of the SADI network to ILL22.

Monitoring a transuranic experiment on D15.

Computerisation of technical drawings for the instruments using the 'AUTOCAD' system (contract with the Reactor Department).

Coordination of the rewiring of the electricity supply network on the instruments (safety improvements).

Assistance with the project for neutron distribution to ILL22 on guides H53, H511, H512.

Measurements of neutron and gamma background levels on the new H5 guides and on the instruments IN14, Gams, IN10C, D3B and D9B.

*Furnace for IN10C.*

# Temperature, Pressure and Vacuum Group

## Standard cryogenics

During 1988 the standard cryogenics laboratory has continued its efforts to maintain and improve equipment and facilities available to the instruments.

The recently designed cryo-furnace (3 K to 600 K) complete with power supply, adjustable support, and liquid level detectors, has been commissioned and is now available on a reservation basis. The D2B cryo-furnace has been modified following this development and the modification of the IN10 cryo-furnace is programmed for the beginning of 1989.

A number of cryostats have now been equipped with automatic cold valves and a further series of these cold valves is in production.

The old type liquid nitrogen and liquid helium level gauges have been replaced by newly designed instruments which facilitate the automatic transfer of cryogenic liquids.

A new helium gas safety valve has been developed and fitted to the liquid helium storage vessels and certain cryostats. Using this valve we have almost eliminated the frequent damage to siphons, improved the safety for users and contributed to the reduction of helium losses.

The consumption of liquid helium at 59,185 litres was almost identical with the figure for 1987. The recuperation rate was slightly down from 89% in 1987 to 87% in 1988.

After taking into account losses and internal use, the cost per litre of liquid helium was 24 F.

Argon gas instead of helium is now used almost exclusively in neutron flight paths, representing a saving of about 50 KF/year.

The total budget for cryogenic fluids exceeded 1.7 MF in 1988, 1.4 MF for liquid helium, 90 KF for argon gas, and 225 KF for liquid nitrogen.

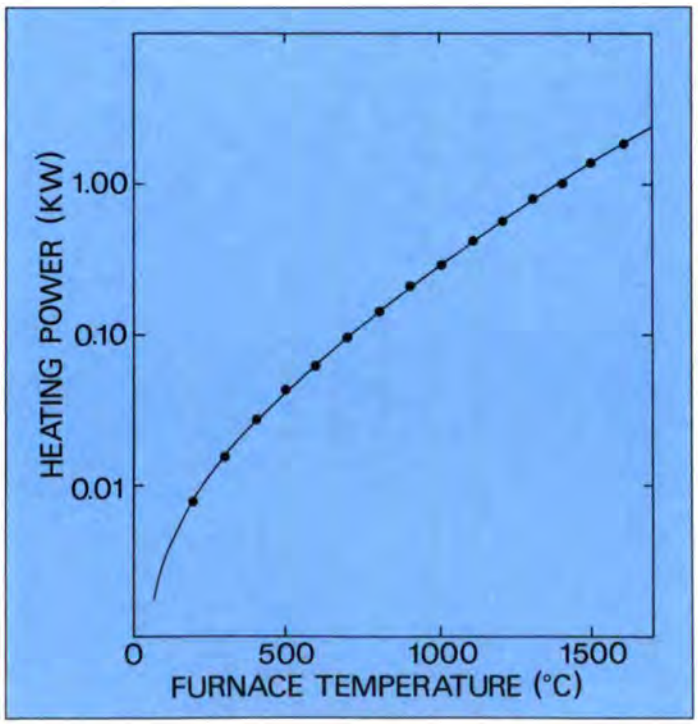



*Figure 103 Relation between input-power and temperature achieved for an ILL standard furnace with an available sample volume of 40 mm diameter and 80 mm height.*

## Vacuum

Apart from maintaining the usual high standard of assistance and maintenance, this group has continued its renovation programme with the acquisition of a number of new pumps and auxiliary equipment. A second batch of single stage rotary pumps have been transformed into two stage pumps and two new helium pumping tables have been built.

Servicing techniques have been improved with the installation of new equipment and the personnel have benefitted from special vacuum courses that they have attended during the last year.

## High temperature

During 1988, 32 high temperature experiments were performed covering 7634 hours of operation. A number of new or modified furnaces has been put into operation. It is worthwhile to mention the new IN10C furnace. This furnace has a large sample volume (up to 70 mm diameter and 80 mm height) and a particularly small outer diameter of 120 mm. This furnace can be used up to 1000°C. For all furnaces a new type of resistor including a better thermal shielding has been developed. As a measure of the quality of this new heating unit a typical curve of the relation between input power and temperature achieved is shown. (Fig. 103). As from September 1st 1988 the role of the scientific animator was taken over by W. PETRY from A. WRIGHT. We would like to thank A. WRIGHT for his excellent support and fruitful animation in the field of sample environment at high temperature.

## High pressure

More than 25 high pressure experiments were performed in 1988. Most of these experiments have also involved low or high temperature environment. Single crystal diffraction and inelastic scattering experiments were carried out on many instruments.

Powder diffraction as well as time-of-flight experiments have also been performed with success.

The 30 Kbar pressure cells are not yet fully operational. Improvements are needed in order to obtain reproducible and reliable working conditions.

The manpower of the high pressure group was reduced by the retirement of one technician who was not replaced: his post had to be given up under the staff reduction programme at the end of the 'deuxième souffle'.

The development of new high pressure cells has however been reactivated now by the fact that an engineer, K. GOBRECHT, is working on a part-time basis in this field.

# Chemistry Laboratory

An inventory has been carried out, and a full list of the standard chemicals and a preliminary catalogue of the general equipment, are now available.

A temperature control unit for the IR spectrometer has been ordered and will be installed soon. The new vacuum line has been improved and powder samples can now be heated under vacuum to 1200°C.

A general safety examination has been made, and it is considered by those responsible that more space is required. Special storage facilities for solvents and acids will be installed by the end of 1988.

A particular effort was made in 1988 to improve communications between the users and the persons in charge. We hope this will continue in 1989, in order to improve the development of chemistry facilities at the ILL.

# Building Maintenance and Modifications Service

The main responsibilities of the Service are the maintenance and improvement of the ILL site, buildings and technical installations (except the reactor) and adaptation of the experimental positions to the users requirements. Therefore the Service has three essential activities in two main directions (site and buildings, experimental positions).

## Maintenance and Repairs

### Site and buildings

The main purpose is to keep in an operational state all the buildings and technical installations (electricity, drainage, gas, heating, air-conditioning, ventilation, handling); the interventions required are carried out with the workshops of the Group or by the placing of external contracts. The Group also supervises the general cleaning of buildings and open spaces and waste removal, and it takes charge of furniture purchases and office or laboratory removals.

### Experimental positions

At the request of physicists or the Central Service the Group executes all work necessary for keeping in order technical installations associated with the experiments.

## Construction, Renovation and Equipment Work

### Site and buildings

At the request of the Directors or in order to improve the running of existing installations, the Group designs and follows up the construction, renovation or modification of buildings, offices and laboratories, and technical installations. In 1988, the principal work has included internal repainting of the ILL 2 building, construction of the ILL 22 extension, finishing of the fire protection for the reactor technical areas in the ILL 4 building, reconstruction of the reactor entrance guard room and creation of new offices for physicists in ILL 4 building.

### Experimental positions

In collaboration with the Central Service, the Reactor Department, and the Instruments and Methods Department, the Group defines and organizes the construction and assembly of the biological shielding and infrastructure required around the instruments in the experimental halls. Major projects carried out in 1988 were completion of the shielding around associated guides for the new cold source, finishing infrastructure for the $n\bar{n}$ experiment and D3B, installation of IN10C and IN14, preliminary work for the installation of IN15 and D22 and participation in the change of the H9 beam tube.

## Technical Assistance to other Groups

### General services

The "Self-Service" workshop and the main sheet metal workshop were used to carry out modifications or production of items which were difficult to sub-contract, or were needed at short notice, or which needed on-the-spot presentation and adaptation. The Group was also involved in provision of logistical support for special visits and events in the life of ILL.

### Services for external organisations

The Group carried out maintenance of the building and the technical installations of the EMBL outstation. In collaboration with ESRF it prepared the necessary site modification for the synchrotron buildings; the major work in 1988 was modifications to the road layout and the main site fence, construction and transfer of the buildings ILL9, ILL10, ILL14, ILL23 and ILL24. Studies were also carried out for a common library and canteen building and for a common site administration.

The Group was also involved in the installation of the drainage system associated with the EDF dam on the river Isère and in the studies for the supply of fluids to the ESRF (water, electricity and superheated steam for heating).

# Fundamental and Nuclear Physics

PN1 Fission product separator (LOHENGRIN) on beam-tube H9 (H. R. Faust, J.P. Bocquet, R. Brissot, I. Gartshore).

PN2 Beta spectrometer (BILL) on the vertical beam-tube V3 (B. Krusche, S. Judge, K. Schreckenbach).

PN3 Three curved crystal spectrometers (GAMS 1,2,3) and one flat crystal spectrometer (GAMS 4) on the throughgoing beam-tube H6-H7 (H. Börner, S. Robinson, P. Schillebeeckx, S. Dewey, R. Oliver).

PN4 Ge(Li) pair spectrometer on beam-tube H7 (S. Robinson, R. Oliver).

SN5 Ultra-cold (UCN) and very cold (VCN) neutron source with 2 beam positions on the inclined beam-tube IH3 (P. Ageron, W. Mampe, A. Beynet).

SN7 Cold polarized neutron beam at end position of guide H14 (K. Schreckenbach, D. Dubbers).

PN8 Fission product coincidence spectrometer (Cosi Fan Tutte) (P. Geltenbort, T. Manning)

H17 Cold neutron guide with liquid helium UCN source (P. Ageron, I. A. Kilvington).

H18 Cold neutron guide (W. Mampe).

H22 Thermal neutron guide: neutron induced particle emission (H22D), (J. P. Bocquet); prompt gamma activation analysis (H22E), (S. Robinson, R. Oliver); $\gamma, \gamma$ angular correlations (H22 F), (S. Robinson, R. Oliver).

TGV Vertical VCN guide in cold source connected to Steyerl turbine producing UCN (Niveau D), (P. Ageron, W. Drexel, W. Mampe, A. Beynet, A. Steyerl).

$n\bar{n}$ Neutron-antineutron oscillation experiment at the cold neutron beam H5 (D. Dubbers).

*View of the central part of the GAMS4 spectrometer. The γ-beam originates from the left side in the picture. It is diffracted by two Si-crystals which share the axis of rotation of the two angle interferometer arms. The crystal on the right side shares additionally the rotation axis of a 24 sided polygon which allows - in conjunction with an autocollimation system - the absolute angular calibration.*

## Fission Research

### PN1

At PN1 the beam-tube H9 was exchanged during a long reactor shut-down in spring 1988. The work was carried out by the Reactor Department and EDEX. At this opportunity several parts of the spectrometer were also revised such as the target changing facility, the pumping system, safety shutter etc. Furthermore, a collimator system was designed and constructed to be inserted between the analyzer magnet and the condenser of the spectrometer. The system allows tests on the optical performance of the beam and a narrow beam collimation for high resolution measurements.

A major effort was undertaken in 1988 to install a new high quality data acquisition and inspection system, both in soft- and hardware. An Atari computer was connected to a fast bus system to allow for the collection of 6 parameter list mode data. A convenient software package has been written to control on-line a matrix of 1 Mbyte as well as the incoming single spectra. The system is very fast (data rate 8 KHz in 6 parameter mode) and has proved its reliability in a 6-month running period without failure. The purchase of a μVax for PN1 will make the treatment and reduction of list-mode data at the instrument possible.

### PN8

The coincidence fission fragment spectrometer "cosi fan tutte" was operating smoothly throughout the year. The investigations on various fissile targets are outlined in the scientific section of the report.

## Spectroscopy

### PN2

The high resolution electron spectrometer BILL was running continuously throughout 1988.

For standard conversion electron measurements, the on-line control and the data treatment were improved by the installation of new graphics hard and software.

For high resolution measurements an additional system of movable slots was inserted into the beam-tube close to the exit of the first magnet. This system allows for a narrow collimation of the beam and hence better resolution, in particular at low electron energies, where remanences in the magnet play a major role. Of course the gain in resolution is at the detriment of intensity.

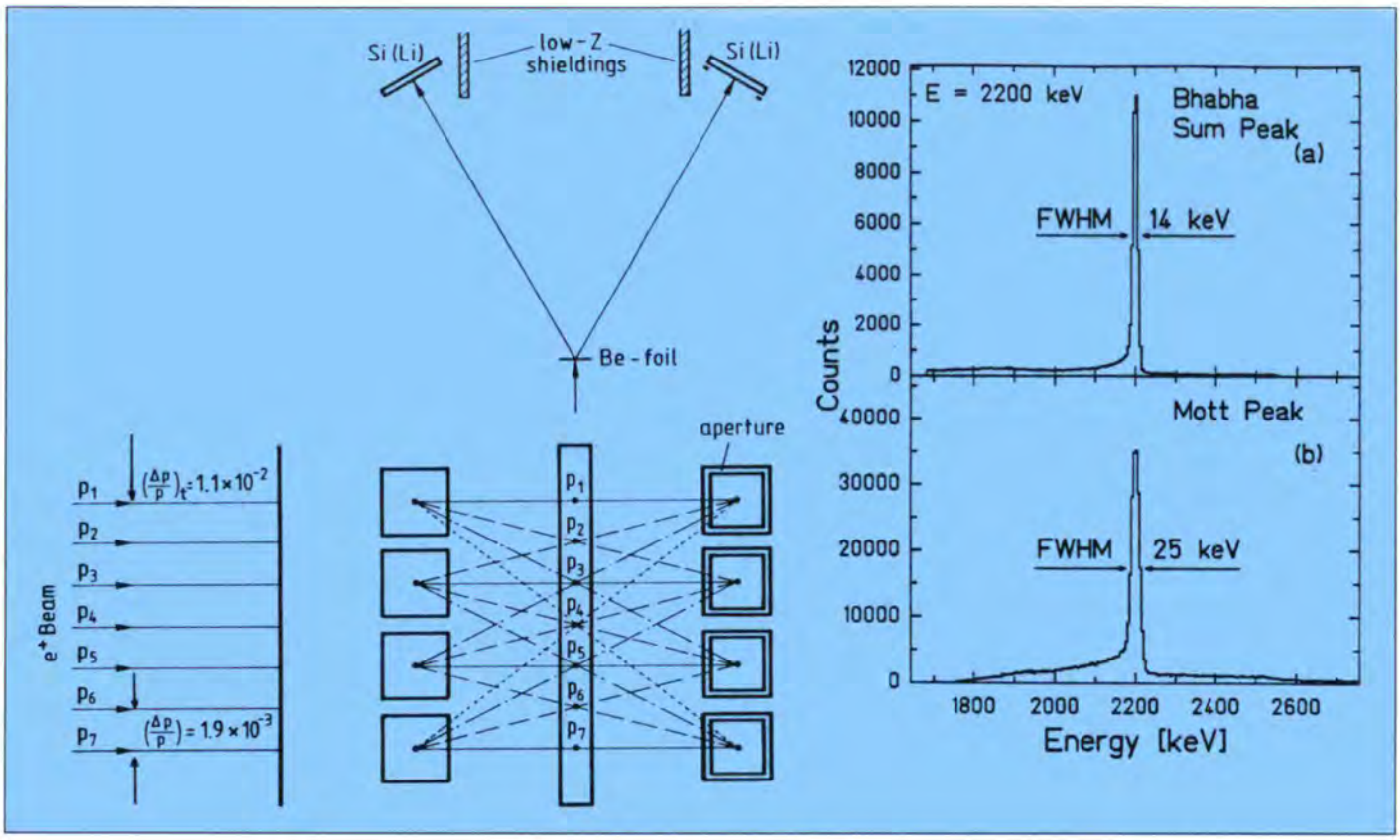



*Figure 104 Experimental set-up for the investigation of Bhabha and Mott scattering. The beta spectrometer focuses the $e^+$ beam onto a Beryllium foil placed as scatterer in its focal plane. The scattered particles ($e^+$, $e^-$) were detected by two arrays of four Si (Li) detectors. Spectra recorded in 165 min at a beam energy of 2200 keV are displayed in the insets (a) and (b). The width of the Mott peak reflects the total momentum spread ($\Delta p/p$) = 1.1 x $10^{-2}$ on the Be foil, while the information on the scattering point for Bhabha events reduces the observed momentum window to 1.9 x $10^{-3}$ and hence leads to a narrower line width for the sum peak.*

In 1988 again much effort was made to improve the experimental set-up for the measurement of the excitation function in $e^+e^-$ (Bhabha) scattering, searching for possible resonances. The intensity of the in-pile positron source at BILL was further increased by about 50% and provides now 1.2 x $10^6$ $e^+$ per second in a 10 x 100 $mm^2$ segment of the focal plane of the spectrometer. This value is almost constant over the interesting energy range between $E_{e+}$ = 2 MeV and 3 MeV. The new positron source is composed of a 50 x 130 x 2.3 $mm^3$ titanium plate covered in the center by a 50 x 50 x 0.25 $mm^3$ platinum foil.

As in the previous source the positrons stem mainly from pair production in the absorption of high energy $\gamma$-rays produced by the $^{48}Ti(n,\gamma)^{49}Ti$ reaction. The platinum contributes only marginally to the high energy $\gamma$-ray flux, but acts as an efficient converter, due to the high external pair creation cross-section of high nuclear charge material.

The weakly bound electrons in the low-Z element Beryllium were chosen as scatterer to minimize the Doppler broadening of a possible resonance. For free atoms this broadening, caused by the motion of bound electrons, amounts to 13 keV FWHM in Beryllium, but to ~ 60 keV for Thorium (laboratory system). From the measurement of the total Compton profile of metallic Beryllium a broadening of about 27 keV was derived, which dominates the energy resolution in the present experiment.

For the detection of the scattering events a new scattering chamber was constructed. The scattered particles from the 4.6 $mg/cm^2$ Beryllium foil in the focal plane were recorded with an 8 detector device (Fig. 104). The high resolution Si(Li) detector (20 x 20 $mm^2$, 2 mm effective thickness, cooled) were arranged in two arrays symmetrically to the beam. They are movable under vacuum from outside the chamber for adjustment of the geometry.

Single events and coincidences between all detector combinations were recorded in list mode. The figure illustrates that for coincidence events from opposite detectors the scattering point at the Beryllium foil can be reconstructed and hence the incident energy for the event determined better (momentum dispersion of the spectrometer $\Delta p/p$ = 1.5 x $10^{-4}$ per mm). An example for energy normalized spectra for single events (Mott scattering) and coincidence events (Bhabha scattering) is given in the Fig. 104. Since for the latter 7 incident energies are discriminated, but measured simultaneously, possible systematic errors are efficiently reduced. Time-correlated fluctuations are smeared out over the 7 points when data from corresponding energies are summed up.

Even though this experiment has already yielded the most stringent limit for possible resonances in Bhabha scattering (see scientific section) further improvements are feasible. The measurements set a minimum lifetime for a hypothetical object decaying into $e^+e^-$ pairs of 3.5 x $10^{-13}$ s.

Therefore it is envisaged to distinguish between $e^+e^-$ events from inside and after the scattering foil. Events of the latter type cannot result from conventional Bhabha scattering. First tests of a new experimental set-up are currently under way.

### PN3/4

Experiments at the in-pile (n,γ) facility were shared between the bent crystal spectrometers GAMS 1,2,3, the double crystal spectrometer GAMS4 (see photograph) and the pair spectrometer PN4, leading to a heavy demand for this beam position. Various improvements were implemented at the instruments.

At the bent crystal spectrometers the data acquisition system has been further developed and can now be used directly for the different spectrometer crystals (Quarz and Silicon). More diagnostic features and a facility to monitor the spectrometer from a remote terminal were included.

For use with the Silicon crystals a new crystal bender has been designed and is currently being tested. This mechanical device should permit the precise bending of a rectangular crystal. It is envisaged to obtain the same high resolution as with the previously constructed triangular crystal bender, but with a significant increase of the useful reflecting area on the crystal.

For GAMS 2/3 a new casemate was constructed for additional shielding against the radiation background from neighbouring instruments. For the same reason the shielding for GAMS4 was improved considerably.

The spectrometer GAMS4 was equipped with an additional beam shutter which is controlled by CAMAC. This shutter allows for an automatic change of the position of the spectrometer without risk for the gamma detector. Such changes are necessary to measure in different modes of reflection (dispersive, non-dispersive).

The Germanium detector at PN4 has shown a severe degradation in resolution due to excessive neutron radiation damage. The specially shaped detector is used for measurement of high energy primary gamma-rays in the pair spectrometer mode. A new detector has been ordered and delivery is expected in December 1988. This new detector has a fast baseline restore preamplifier allowing for much higher counting rates to be treated without loss in resolution.

### SN5

The SN5 ultra-cold neutron beam has been used as a test facility for various preparatory experiments.

### H17

Work on the superthermal Helium source for ultra-cold neutrons has been continued by the Berlin group and the ILL. A statistical chopper has been designed and put into operation at the source exit in order to study the upscattering process of ultra-cold neutrons in suprafluid Helium-4. A Helium-4 purifier is close to completion.

### H18

The 20 Å beam H18 has been used for the measurement of interference patterns with both absorption and phase shift gratings as to be used for the new very cold neutron interferometer (Vienna-Munich).

### SN7

The SN7 beam was used for experiments with continuous, chopped and polarized neutron beams. Experiments comprise cross section for neutron emission in fission (MOL/GEEL), parity violation in fission (Tübingen) and a neutron life time measurement (SUSSEX, N.I.S.T.).

### $n\bar{n}$

The installation of the $n\bar{n}$ experiment was finished in 1988 and the experiment has started to run.

### S50

The long baseline experiment for a precise determintion of $h/m_n$ became operational at the monochromatic thermal beam S50 (PTB Braunschweig).

Coordinator: K. Schreckenbach

# Fundamental Physics

A variety of fundamental physics experiments were performed in 1988 on different neutron beams: VCN beam and UCN beams from the neutron turbine (TGV on Niveau D), the original UCN source SN5, the cold neutron beams H17, H18 and SN7, the new cold beam for $n\bar{n}$ and several dedicated experiments at thermal beams (H22, S50). The results of the experiments are summarized in the scientific section for College 3. Some comments are given here as to technical developments and use of the beams.

### VCN beam and UCN beams at level D

The technical development at this very cold and ultra-cold neutron facility on level D of the reactor is described in the section Special Instruments and Experiments. Fundamental physics experiments were carried out on the EDM of the neutron (Harvard-ILL-Rutherford-Sussex-Washington collaboration) and on the neutron lifetime with a fluid wall neutron bottle (ILL).

A neutron gravity monochromator was constructed and Fermi potentials for material walls were measured (SUSSEX). A long base line neutron interferometer for 100 Å neutrons is currently installed (Vienna-Munich).

# Three-Axis Spectrometers

IN1 3-axis and beryllium-filter spectrometer on the hot source beam-tube H8 (B. Dorner, H. J. Lauter, J. P. Varini).

IN3 3-axis spectrometer on the thermal guide H24. (H. J. Lauter, L. Cussen, V. Frank, R. Arthaud).

IN8 3-axis spectrometer on the thermal beam-tube H10 (C. Vettier, M. Alba, A. Brochier).

IN12 3-axis spectrometer on the cold guide H142. (S. M. Hayden, H. Godfrin, D. Puschner).

IN14 3-axis spectrometer on the cold guide H53 on the second cold source (R. Currat, M. Alba, W. Kaiser, A. Brochier).

IN20 3-axis spectrometer for neutron polarisation analysis on the thermal beam-tube H13. (P. Frings, J. Martinez, P. Flores).

## General

Despite a reduced staff (P. Frings has moved to the University of Amsterdam), all three-axis instruments have offered reliable performances to ILL users. A recent survey has shown that around 88.5% of the reactor beam-time was allocated to accepted proposals over the last two years.

1988 has been the last year of general user service for IN3. In the future this instrument may be either transformed into a Special Instrument or shut down.

*Bottom part of the super-conducting magnet for 3-axis spectrometers (Max. field: 6 Tesla, temperature range 1.5 K < T < 300 K).*

## Instruments

### IN1 Hot source 3-axis spectrometer (H8)

The instrument was operated over the scheduled period without any major problem. As it had already been done last year, the amount of beam-time available was slightly extended at the expense of the Be-filter instrument. Nevertheless the overdemand factor was larger than four.

Peripherals and standard TAS programs for instrument control and data analysis have been implemented.

### IN1 Be-filter (H8)

The Be-filter instrument has been operating very reliably. The Be-filter (5 meV resolution) is used in combination with the (200) and (220) Cu monochromators. The Be graphite combination filter with a resolution of less than 2 meV usually requires the use of the Cu (331). A high transmission graphite filter is being built.The operating time has been reduced due to the heavy overload on the three-axis instrument IN1.

### IN3 Three-axis spectrometer on the thermal guide (H24)

Only minor changes were made during 1988 and the instrument is running perfectly well in its present state. In 1989 the instrument IN3 will become an S-instrument to be used by external user groups.

### IN8 Three-axis spectrometer on the thermal beam (H10)

IN8 has continued to run reliably over the whole scheduled period. The four monochromators have been widely used together with the optional in-pile Soller collimators. The mechanics is very reliable and no drift in angular positions has been observed. Some improvements such as reduction in background (1 cpm at present) and computer controlled analyzer curvature may be implemented during the next period.

IN8 is one of the most oversubscribed instruments at the ILL. As such it has contributed to major research investigations on high $T_c$ super-conductors, heavy fermions, phonons in bcc metals, ...

## IN12 Three-axis spectrometer on the cold guide-tube (H142)

At the beginning of the year, the computer system and some associated electronics were replaced. IN12 is now equipped with a PDP11/73 computer and closed-loop/DC motor system, the new electronics allows many new parameters (e.g. guide fields and flippers) to be controlled directly by the computer. The new system worked well throughout the year.

The guide carrying neutrons to IN12 is designed such as not to transmit higher energy neutrons: we would otherwise have higher order contamination above $K_i$ = 1.55 Å$^{-1}$ where the Be-filter cannot be used. Recent measurements have shown that the higher order contamination in this range of $K_i$ is very low: 2 % for $K_i$ = 1.6 Å$^{-1}$ and less than 1% for $K_i$ = 2.0 Å$^{-1}$.

## IN14 Three-axis spectrometer on the horizontal cold source (H53 guide)

The construction of IN14 in the Hall d'Essais and on site continued on schedule. The first neutron tests for the PG(002) monochromator and primary spectrometer took place in June. The monochromatic beam was calibrated with respect to flux, homogeneity, and higher-order contamination, for several incident neutron energies. Flux figures are comparable with IN8 at 13 meV, while in the cold neutron range (2 meV < $E_i$ < 5 meV), a 3-fold gain is achieved over IN12. Further tests were carried out in October, in order to calibrate the complete spectrometer with regard to resolution and background (1 to 2 cpm under normal inelastic scattering conditions).

At the time of writing, the spectrometer can only operate in the constant-$K_i$ mode. It is hoped that the fully-automated constant-$K_f$ mode is available at the February start-up. The first half of 1989 will be devoted partly to scheduled experiments and partly to instrument developments. These will involve the implementation of the polarized beam option (supermirror bender and Heusler analyzer), the construction of a continuous-wavelength higher-order filter and the installation of a second monochromator crystal Si(311).

## IN20 Three-axis spectrometer with polarization analysis on the thermal beam (H13)

IN20 has been fully operational during 1988. The main improvement was the new Helmholtz coil with a three-fold symmetry about the vertical axis. There are three windows of 110° to access the sample. Magnetic field (up to 15 0e) can be applied to the sample in any direction with the help of four power supplies which are computer-controlled. A remote control PG filter has been developed to improve the flexibility of the instrument during sample alignment. The analyzer mounting has been modified in such a way that various analyzer crystals from other TAS instruments can be used for experiments without polarization analysis.

The absolute sensitivity of the instrument was measured with a Vanadium sample at $K_i$ = 2.662 Å$^{-1}$ ($\alpha_1$ = open, $\alpha_2$ = 40', $\alpha_3$ = $\alpha_4$ = open) with a PG filter. With the PG(002) monochromator and analyzer, 1 mm$^2$ total scattering cross-section gives 70 n/s in the detector; with Heuseler crystals this reduced to 6 n/s.

Further developments will probably focus on miniaturization of diaphragms to be mounted inside guide-fields, and better magnetic shielding of the flipper placed before the sample.

Coordinator : C. Vettier.

# Time-Of-Flight, High Resolution and Diffuse Scattering

IN4 Time-of-flight spectrometer on thermal tube H12 (A. Murani, H. Mutka, A. Dorn (technician)).

IN5 Multi-chopper spectrometer on cold guide H16 (G. Kearley, F. Rieutord, H. Blank, S. Jenkins (technician)).

IN6 Focussing TOF spectrometer on cold guide H15 (A. J. Dianoux, R. White, S. Jenkins (technician), Y. Blanc (technician)).

IN10 Backscattering spectrometer on cold guide H15 (B. Frick, S. Mahling-Ennaoui, P. Joubert (technician)).

IN10C Backscattering spectrometer at the Horizontal Cold Source (project) (A. Magerl, J. L. Coquin, Y. Blanc (technician)).

IN11 Spin-echo spectrometer on cold guide H15 (B. Farago, J. F. Legrand, J. Bauchat (technician)).

IN13 Backscattering spectrometer for short wavelengths on the thermal guide H24 (W. Petry, J. Williams, J. F. Barthélémy (technician)).

IN15 High-resolution spin-echo spectrometer for long wavelengths (project) (F. Mezei, D. Richter, C. Lartigue, F. Douchin, J. F. Barthélémy (technician)).

D7 Diffuse scattering instrument with polarization analysis on cold guide H15 (O. Schärpf, W. Just, R. Rebesco (technician)).

D11 Small-angle scattering diffractometer on cold guide H15 (P. Timmins, A. Wright, R. May, R. Baker (technician)).

D17 Low Q, low-resolution diffractometer on cold guide H16 (A. Rennie, J. Torbet, R. May, M. Cruz (technician)).

D22 Project of small-angle scattering diffractometer on the Horizontal Cold Source (R. May, M. Thomas).

Group Engineer: F. Douchin

## IN4 TOF spectrometer on thermal beam H12

The single monochromator-chopper configuration of the instrument has been working reliably and has replaced the aging double rotating crystal version for almost the whole year except for only one cycle. The main reasons for this evolution lie in improvements of the instrument performance in terms of background. Additional shielding around the monochromator position and incorporation of a third chopper (although limited to speeds less than 9600 rpm) practically eliminated the pulsed background associated with the fast neutron burst originating during the open time of the first chopper.

Now, in the most frequently used conditions, i.e. incident energies of 17 and 68 meV, the background level is less than for the double crystal system. A decision was taken to shorten the flight box to 2 m during the shutdown at the end of the year. This will increase the counting rate by a factor of around three.

At the moment the continuing modifications are done on test basis and the instrument, unfortunately, is not optimized nor flexible with respect to variation of incident energy. It is expected that these efforts will show the way for a versatile TOF instrument. Preliminary work, for example a design for an evacuated flight box and the evaluation of the possibility of incorporating a Brillouin scattering option, is being carried out at present.

## IN5 Multi-chopper spectrometer on cold guide H16

Following several years of spurious electronic faults IN5 has been completely rewired. New cable-tunnels have been dug to separate IN5-wires from the general melee of the utilities tunnels. Grouping of detectors into columns is now made inside the detector housing giving a considerable reduction in cable lengths and the removal of the detector-grouping control and its cabinet.

The chopper-system has been mechanically reliable and now runs with only forced air-cooling, all water-cooling having been abandoned due to problems with differential expansion. Some residual electronics problems persist.

The final addition of detectors has been made and 80% of the available surface is now covered with a patchwork of detectors which are either: short-type IN5, long-type IN5, IN6-type, or old D11-type. Cadmium collimators on the detectors have been removed leaving 0.8° collimation on all except the small-angle detectors which remain at 0.2°. The fixed detector-arrangement has made a more careful investigation into the background worthwhile with the result that the beam-off background is now 5 Hz for the sum of the entire detector-bank which is the same as it was before with half as many detectors! With the beam on and a cadmium "sample" the corresponding background is now 6 Hz.

The first Brillouin-scattering was successfully made using a compressed nitrogen-gas sample (despite the ailing electronics). Alignment of the D11 multidetector and its beam-stop are crucial for these experiments and long-term arrangements are being considered. A high background which may have been due to impurity gases in the secondary flight-path requires more investigation. At least two more experiments of this type are planned for 1989.

Following the Brillouin-scattering experiment permanent oxygen-analysis of the sample-area box and the secondary flight-path has been installed. With the oxygen levels below 0.1% the measured resolution-function of the instrument does not deviate significantly from triangular above 1/4000 of the peak-height. The improved signal-to-noise ratio and clean resolution-function have recently been exploited to measure tunnelling peaks due to 100 ppm of hydrogen in metals, and also of 3 mg of urea in a 28 g argon matrix.

For 1989 we hope to replace our amnesic PDP11 computer by a micro-Vax.

## IN6 Focussing TOF spectrometer on cold guide H15

IN6 has worked satisfactorily during this year with 42 experiments performed during the 5 reactor cycles. The very low background of the instrument has allowed us, for example, to measure precisely the phonon density of states (PDOS) of the new high $T_c$ superconducting oxides up to quite high energy transfer. Specifically the changes in the PDOS with oxygen concentration have been studied in the $YBa_2Cu_3O_{6+x}$ series.

At the beginning of the year the data acquisition system has been changed to a micro-Vax workstation. The changeover lasted only two weeks during a normal reactor shutdown and the restart was trouble-free. At the present time the only important improvement in the software is the control of the temperature of the cryostat, but data display and data transfer are much faster. It is envisaged to develop 3-D display programs using the colour display of the workstation. This coming year should see the change-over from the old CAMAC time-of-flight electronics to VME, which will permit, among other things, a variable channel width across the time-of-flight scale.

## IN10 Backscattering spectrometer on cold guide H15

The data acquisition on IN10 was changed definitely from the CAMAC/PDP11 system to a VME/μVAX electronics in 1988. After a parallel and successful data acquisition on both systems in the first cycle of the year, the VME/μVAX configuration took over the control in the second cycle. Finally, during the summer shutdown the PDP11 was completely removed and the new configuration is operating since then without any major problems. A dramatic improvement was also achieved concerning the on-line data treatment. New and efficient programs using menu-options and the graphic possibilities of the "GPX working station" allow on the same terminal simultaneously an easy control of the current measurement, including plotting capabilities and raw data treatment. Data from elastic window scans are now stored on a data file and can be visualized on the graphics screen. Furthermore an ETHERnet has been installed allowing now for a remote instrument control.

A new furnace for temperatures up to 1300 K was added to the joint IN10/IN10C equipment. On all cryostats automatic cold valves have been installed as well as automatic liquid nitrogen filling facilities.

## IN10C New backscattering spectrometer on the 2nd cold source

The construction of the primary part of the instrument has rapidly progressed during 1988. The first graphite deflector with anisotropic mosaicity has been aligned and is already mounted in the beam of H53 on its complete orientation mechanism. Also the primary shielding of the deflector is about to be finished, and the focussing neutron guide with its support is ready for installation. In addition the Tanzboden floor has been laid in 1988. All the major components of the sample area as well as the detector and the Doppler drive are due for early next year. At this time a first version of the electronics and computer system with a VME/VAX configuration will be operational. At present, the construction effort is concentrated on the second deflector/chopper system with 120 graphite crystals made at the ILL, on the shielding of the secondary spectrometer and on the analyzer system.

Before the summer shut-down we were able to perform first neutron tests at the future position of IN10C on H53 with a set-up which contained all the essential parts of the primary spectrometer of IN10C: the final first deflector, the focussing guide, a mock-up of the second deflector and a spherically shaped Si-monochromator. These tests enabled us to finalize all the design parameters of the instrument. In addition, beam properties such as higher order contamination, beam divergence and neutron flux were measured at various points of the instrument.

## IN11 Spin-echo spectrometer on cold guide H15

In 1988 there was a high demand for beam-time on the Neutron Spin-Echo (NSE) instrument for the study of the glass transition of different materials. This is partly due to the special feature of the NSE of measuring directly the intermediate scattering function S(q,t) and partly because the dynamical range measured in a single shot experiment is quite large compared to other instruments (the dynamical range is quite vague but we can define it as the ratio of the longest and the shortest Fourier time of the NSE or the maximum energy transfer and resolution of a backscattering or TOF instrument). In NSE the limitation on the long time side is the inhomogeneity of the magnetic field, and on the short time side a minimum field in the precession coils (≈ 1Oe) is required to maintain the beam polarization. Already the earth's magnetic field and the stray field of the polarizer-analyzer gives a resolution of 5 x $10^{-3}$ ns or 130 μeV.

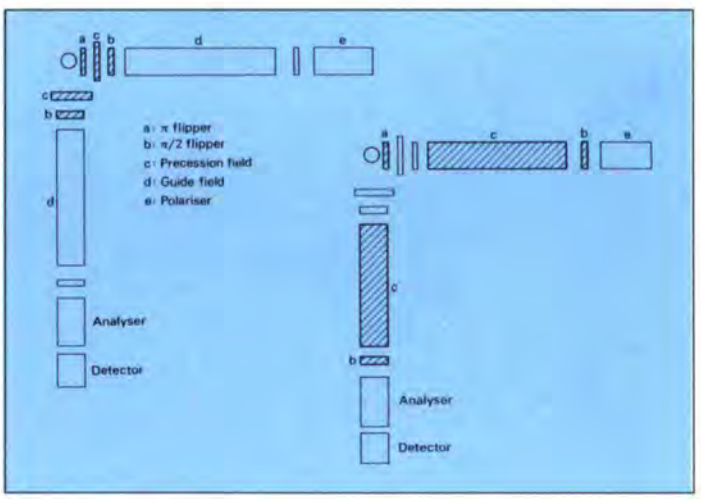
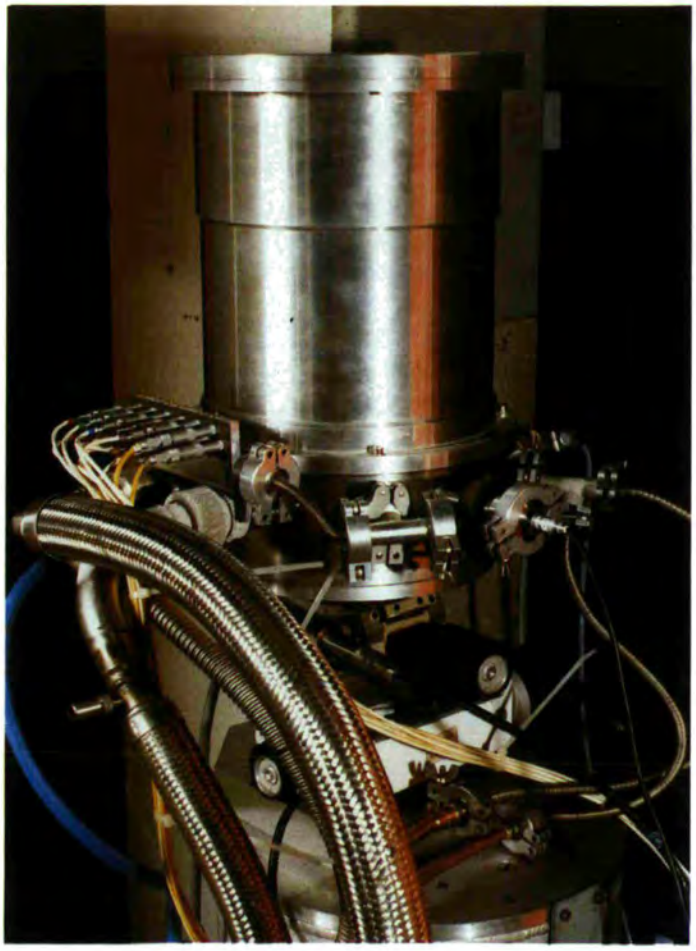
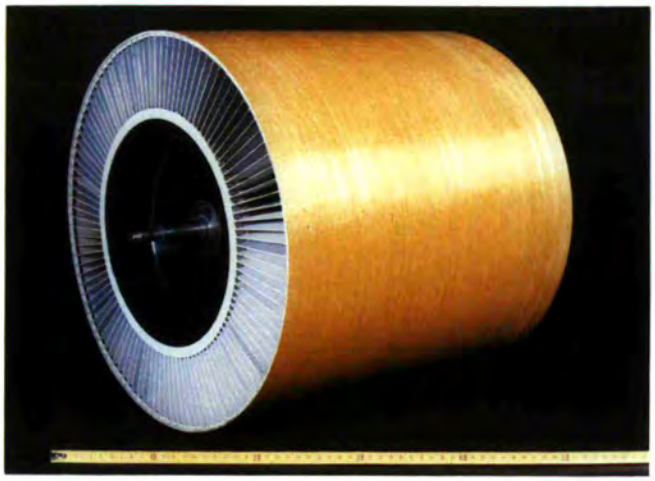


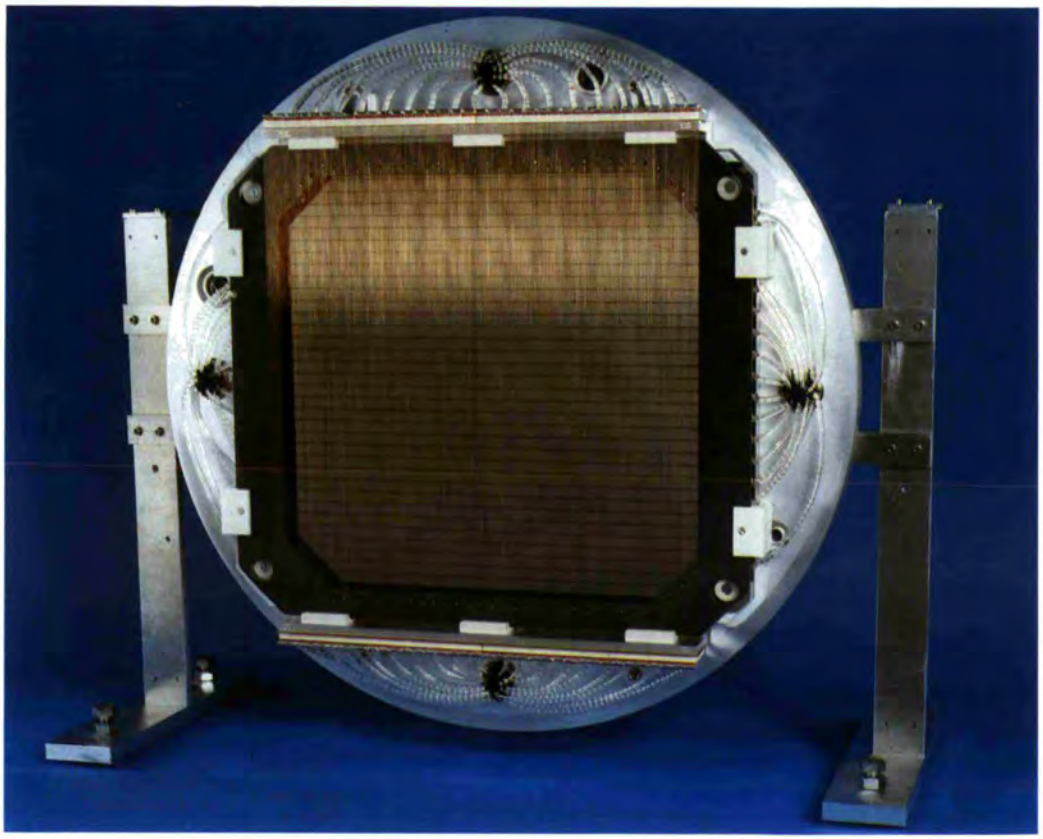

*Figure 105 Schematic layout of the double NSE set-up.*
*a) The inner part is used as an NSE and the outer part ensures only a guide field between the polarizer (analyzer) and the π/2 flippers.*
*b) The inner part is switched off and the outer part is used to measure with the best possible resolution.*



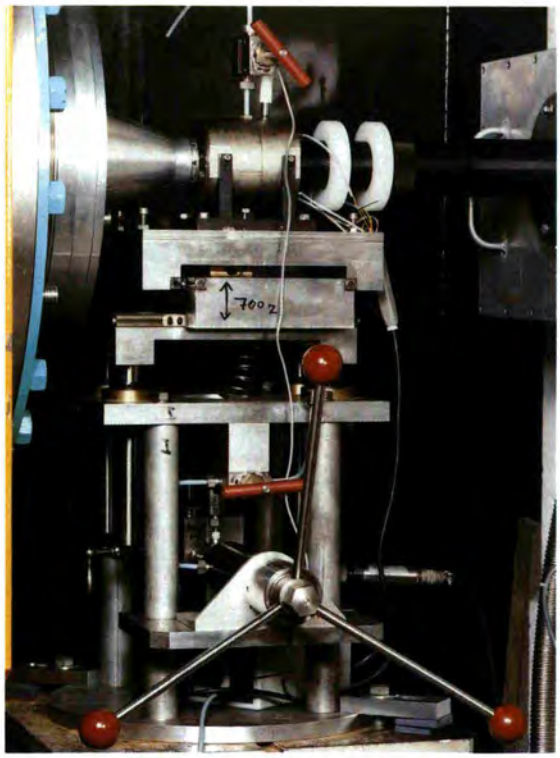


▲ *Velocity selector for polarized neutrons on IN15.*

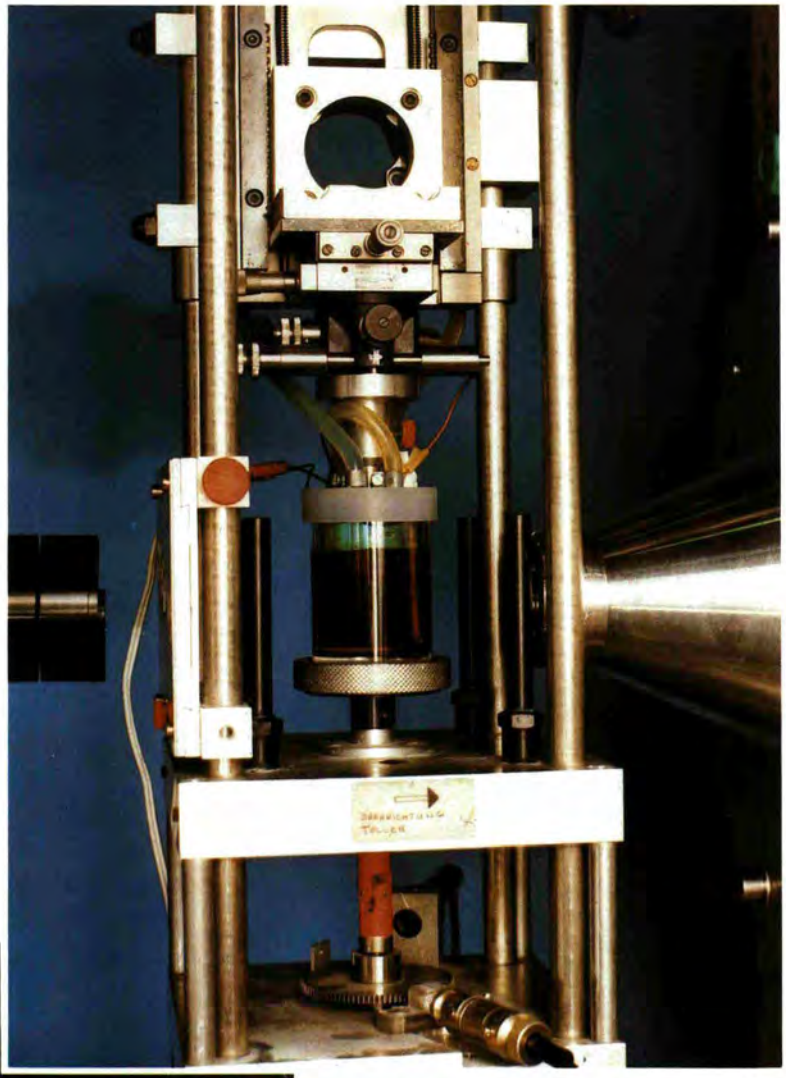
◀ *Cryofurnace for the monochromator crystal of IN13 (temperature range: -190 < T < 450°C.*



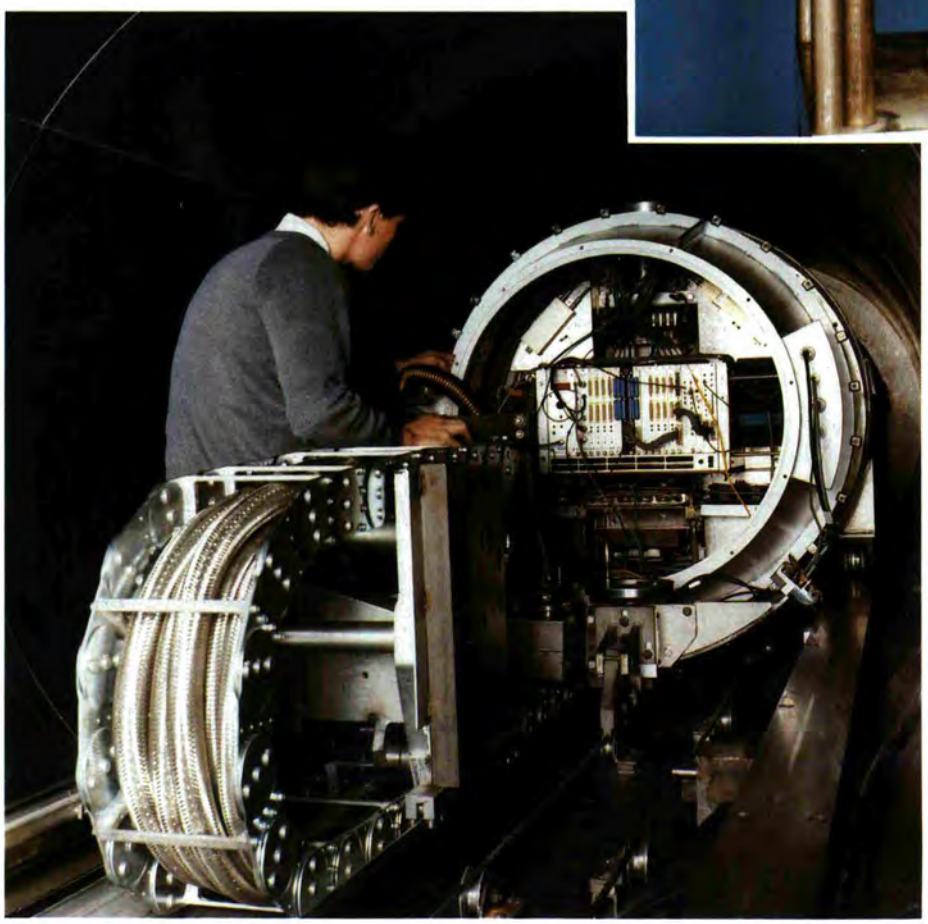
◀ *$^{3}$He multidetector (32 x 32 channels) for IN15.*



▲ *High pressure cell with sapphire windows for the SAS camera D11.*
*(Pressure range 0 < P < 4 kbar).*



▲ *Couette-type shear cell for SAS experiments with liquid systems in a hydrodynamic field at the SAS cameras D11 and D17. Range of shear gradient 0 < $Gs^{-1}$ < 12000.*



◀ *View inside the vacuum tube of D11 showing the detector electronics.*

In a recent experiment with F. Mezei, a solution was found, i.e. to shorten the instrument or, as a practical realization, to transform it into a double NSE instrument. The outline can be seen on Fig. 105. A second pair of $\pi/2$ flipper is placed close to the sample and a simple pair of coils on both sides of the $\pi$ flipper varies the precession field. For the measurement of the short times we use the inner spin-echo and for the long times the outer NSE is used (Fig. 105b). One can switch back and forth without modifying the configuration (wavelength, monochromatization, collimation) and the sample stays under identical conditions (e.g. temperature)

A dynamical range of 640 was reached for the $Ca_{0.4}\ K_{0.6}\ (NO_3)_{1.4}$ sample (Fig. 106). As the measurement was carried out at 90° scattering angle where the magnetic field of the iron carter of D11 seriously limits the use of the maximum precession field. The latter will be replaced, thus allowing for the extension of the dynamical range above 1000.

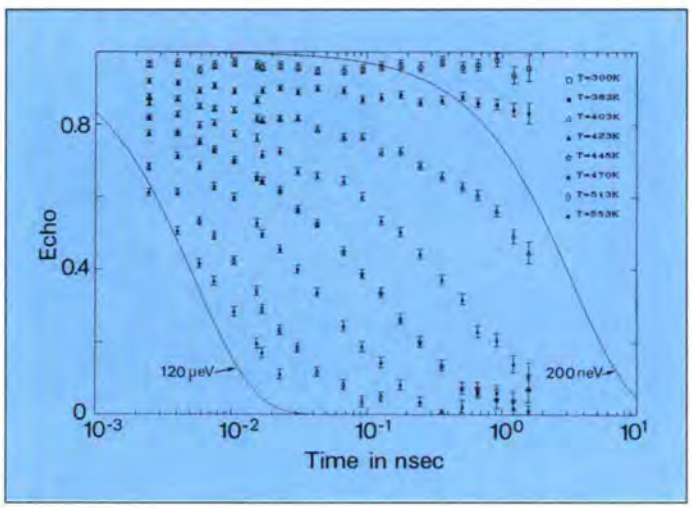



*Figure 106 NSE data as measured with this double NSE. The full curves correspond to Lorentzian line-shapes with 200 neV and 120 μeV (HWHM) decay.*

## IN13 Backscattering spectrometer for short wavelengths on the thermal guide H24

In 1988 IN13 operated in a very stable way. The new instrument software gave excellent performance. For the display of spectra a special version of the programme R is available on the instrument computer. Transfers to the central computer are now fully automated and take roughly 2 s for a whole set of spectra.

The user's trend to demand less time for tunnelling experiments has been continued. Most of the experiments performed dealt with the dynamics of the glass transition in a large variety of glasses. IN13 is particularly suited for these experiments because it offers an interesting combination of high-energy resolution, rather large dynamical range and the unique feature of large Q. A new application of IN13 in the field of coherent critical scattering is worth mentioning. In these experiments coherent quasi-elastic scattering is measured at typically large Q. Experiments on $C_{20}F_{42}$, (the oligomer of teflon), on teflon itself and on $KBr_{1-x}KCN_x$ have been performed.

## IN15 High resolution NSE spectrometer on the horizontal cold source

The 16 m long polarizing guide (glass coated with one GdTi antireflecting layer and one FeCo magnetic layer) was mounted in February with air-cooled coils wound on the housing. Neutrons were available at the exit of this guide in May. The beam was then characterized by TOF and polarization analysis measurements. It was shown that the full magnetization of the FeCo layer can be obtained with a current pulse giving at least a mean pulsed field of 600 Oe instead of 200 Oe as foreseen. Due to the strong remanence of the magnetization of the FeCo layer a few Oe are sufficient to maintain the polarization. Although we have found unexpected losses at large angles, the intensity within the usable divergence at the sample position on the instrument ($\pm 0.5°$) is close to the expected one.

A new velocity selector has been developed at the ILL for polarized neutrons. Its construction is in an advanced stage and is similar to the prototype, now in use on D17, with helical lamellae in composite material installed on a mandrel in aluminium alloy. In order to avoid problems of eddy currents due to the magnetic field necessary to maintain the beam polarization, the latter is now being replaced by a mandrel in composite. It will be tested under vacuum and in a magnetic field in early 1989.

Most of the mechanics will be assembled in the guide hall at the beginning of 1989.

The multidetector (32 x 32 cells) was filled with 100% $^3$He and tested with neutrons on the test beam at $\lambda = 2.4$ Å and is ready for calibration on the instrument.

Progress has been made concerning the coating of the focussing mirror. Neutron reflectivity measurements have shown that a supermirror coating cannot be used due to parasitic scattering at small angles.

## D7 Diffuse scattering instrument with polarization analysis on cold guide H15

Nothing special can be reported about this smoothly running instrument.

## D11 Small-angle scattering diffractometer on cold guide H15

1988 has been a year without serious incidents on D11. The complete experimental program was carried out. Some experiments had to be shifted when it turned out that the replacement of the detector amplifiers and supplies introduced some unexplained parasites; we therefore had to return to the original state of the electronics.

A third pump station has been installed for emergency use. It is sufficient to exchange a few plugs in a special cupboard near the pump control unit to switch between the pump stations. The cone between the detector tube and the sample position has been modified for suppressing shadowing effects in the nearest detector positions. A larger diameter window for special sample-environment set-ups is now available as well. The

rotation stage of the sample table has been adapted for remote control by a step motor drive.

Like on D17, the data evaluation programs, including those for anisotropic data treatment, developed for the central Vax computers after shut-down of the Dec-10 in April were copied to the instrument Vax. A second colour monitor, interfaced with an Atari ST520 micro-computer, has been installed for optional continuous on-line monitoring of the detector data with 16 colours; the previous pseudo-2D live display remains available.

An automatic, computer-controlled wavelength change procedure has been installed. It works via a digital-to-analogue converter (DAC) CAMAC unit supplying a reference tension for the REFU selector power supply. First tests of the "watch-dog" system which automatically dials preselected telephone numbers upon failure of control units or of the control program have been performed. D11 possesses a EUROSIGNAL receiver; D11 users can therefore be reached virtually everywhere in case of an instrument alarm detected by the computer, or in case of a computer or CAMAC-crate failure.

## D17 Low-Q, low-resolution diffractometer on cold guide H16

The year 1988 has been of mixed fortune for D17 - over recent years several problems have become apparent that arise from aging of critical components with no effective programme of investment to maintain high standards of reliability. The velocity selector that failed last year has been replaced and this has permitted us to resume use of the full range of wavelengths at 10% resolution. The selector is of a new design prepared at the ILL in which the helical slots are formed by mounting reinforced plastic lamellae on a central mandrel. The use of polymers in this form instead of slotted aluminium disks has greatly reduced the weight and thus simplified the design of bearings. This selector which will serve as a prototype for replacements for several instruments was constructed and tested very quickly once the design had been agreed showing the advantage of close co-operation between the engineering services and the scientists running the instruments. No substantial problems with the selector have arisen since it was installed in the spring. It provides a slightly higher flux than the previous selector.

In October the multidetector suffered damage to the electrodes when a regulated high-voltage supply failed; unfortunately this will take several months to repair. Although a small linear detector has been installed for tests, it has been necessary to suspend the visitor programme on the instrument. Some of the reflection experiments can be performed using this detector and it is hoped that the normal range of experiments can be resumed soon after the reactor start-up in 1989.

Despite the difficulties described above, a large number of experiments have been performed on D17 during the year. Its use for reflection work is on the increase, and the unique possibility of long wavelengths and a large multi-detector have prompted several new experiments. The usual range of smaller developments have been made to both hardware and software. Better monitoring of the instrument during measurements is available, an IEEE bus on the computer allows simple interfacing of visitors equipment. With the disappearance of the DEC-10 computer all programs for treatment of small angle scattering were revised and a complete set, including anisotropic analysis, is now available on the instrument computer.

## D22 New low-Q diffractometer on the horizontal cold source

The project of a new small-angle scattering instrument on the horizontal cold source, D22, has now entered the construction phase. The design of both, the detector tube and the collimation unit, is completed.

The stainless-steel detector tube, with a diameter of 2.54 m, offers minimal and maximal sample-to-detector distances of 1.2 m and 18 m, respectively. It can house a 1 m x 1 m detector a prototype of which is under development at the ILL for an inelastic scattering facility of the Hahn-Meitner Institut (HMI) in Berlin. The D22 detector is planned to have 128 x 128 detection elements of 7.5 x 7.5 $mm^2$. An adaptor flange for temporary use of the smaller D11 spare detector will be provided as well, so that the start-up of the instrument will not depend on the availability of the large detector. An increase of the Q-range covered in any single measurement is achieved by an optional lateral displacement of the detector of up to 50 cm, combined with a rotation around the central vertical axis of the detector minimizing parallaxis effects. The manufacturers plan the installation of the tube in September 1989.

After detailed discussions, a mechanical set-up of the collimation unit identical to that of HMI's (conceived in close contact with scientists and the construction office of the ILL), with the exception of the lengths of the guide drums, has been chosen. All parts are, as far as possible, made of aluminium for providing a clean magnetic environment for IN15 and the $n\bar{n}$-experiment (and to facilitate neutron polarization for D22 in the future). Delivery of the collimation unit is foreseen for July, 1989.

A custom-made vacuum pumping unit, including the control device, has been studied and bought. The design of the remaining parts like the sample environment equipment is based on that of D11 and D17. The accessibility of the sample zone, which will be difficult due to the large detector-tube diameter, will be improved by providing several sample tables on which the next experiment set-up can be prepared outside the instrument and moved into position on air pads when its turn has come.

A Dornier/Braunschweig 10% resolution selector will be tested next year. ILL's own experience with composite selectors (IN15) is improving rapidly, too, and might provide us with an interesting alternative. Life-time considerations lead to think that gadolinium has to be used as absorbing material rather than boron.

The development of an electronic "patch-board" unit in the VME standard has been started by ILL's electronics group.

Coordinators: A. Dianoux
R. May.

# Diffraction Instruments

D1A High resolution powder diffractometer on thermal guide H22 (A. W. Hewat, J. K. Cockcroft, J. Davies).

D1B Two-axis diffractometer with multidetector on thermal guide H22 (C. Ritter, J. L. Soubeyroux, K. Ben Saidane).

D2B Very high resolution powder diffractometer on thermal beam H11 (A. W. Hewat, T. Vogt, J. Davies).

D3B Two-axis polarized neutron diffractometer with lifting counter on hot beam H4 (F. Tasset, P. Feder).

D4B Disordered materials diffractometer sharing the hot beam H8 with IN1B (P. Chieux, J. Rodriguez, P. Feder).

D9 Four-circle diffractometer on the hot beam H3 (M. S. Lehmann, J. Archer).

D10 Four-circle triple-axis spectrometer on thermal guide H24 (C. M. E. Zeyen, T. Brückel, R. Chagnon).

T12 Neutron camera on thermal guide H23.

D15 Four-circle MK6 diffractometer on the inclined thermal beam IH4 (J. Brown, M. L. Vrtis, G. Schmid).

D16 Four-circle MK6 diffractometer on cold guide H16 (G. Zaccai, E. Pebay-Peyroula, J. M. Reynal).

D19 Multidetector diffractometer for protein crystallography on the thermal beam H11(S. A. Mason, J. Chevrier, J. Archer, D. Robinson).

D20 High flux multidetector diffractometer on the thermal beam H11 (J. Pannetier, P. Convert, J. Torregrossa).

DB21 Four-circle diffractometer with PSD for biological macromolecules on the cold neutron guide H15 (M. Roth, P. Metcalf (EMBL), P. Agnes).

S18 Neutron interferometer on the thermal neutron guide H25 (H. Uebbing, G. Schmid).

S20 Neutron topography diffractometer on the thermal neutron guide H24 (J. Baruchel).

S21 Double crystal spectrometer on the thermal neutron guide H24 (C. M. E. Zeyen, R. Chagnon).

S42A Laue Diffractometer (J.C. Marmeggi, W. Drexel).

S42B Laue Diffractometer (J. Baruchel).

## D1A High resolution powder diffractometer on the thermal neutron guide H22

In June 1988, a paper produced on D1A (J.J. Capponi et al., Europhysics Letters, **3**, 1301 (1987)) was reported in the US journal "The Scientist" as being one of the eight most cited of all scientific papers in the previous twelve months. Amusingly, at the time the work was done, D1A was not a regular ILL instrument! As a result, in 1988 D1A was restored to the list of normally supported machines, and was again scheduled for regular experiments by the Science Council. However, we still rely on the support of Professor A. Simon, whose Institute pays the salary of the instrument scientist in return for special beam-time.

We also sell some beam-time for experiments on stress in engineering materials, and groups from Britain, France and Germany are allocated additional time by the College 5 subcommittee for such measurements. This money goes to the general ILL budget in return for normal financial support for the remainder of the experiments.

D1A is an instrument where experiments, longer than the usual 3-4 days possible on D2B, can be performed. As well it has unique advantages for investigating unknown and magnetic structures, since long wavelengths (2 or 3 Å) can be obtained from the neutron guide with little harmonic contamination. The demand for beam-time is however, well in excess of the time available, and we would like to make the instrument more efficient by adding more detectors.

## D1B Two-axis diffractometer with multidetector on the thermal guide H22

After major changes on the instrument which took place in 1986 and 1987, 1988 was an exceptionally good operating year for D1B. Although there were only 5 cycles free for scheduling, not less than 56 experiments were performed on the machine. As a consequence of the increase in neutron flux and the facility to change between different sample environments, the time needed to perform a successful study on D1B is continuously decreasing. While in 1986 the average period of an experiment was nearly 5 days, it is down to less than 3 days nowadays. This means of course more work for the instrument responsibles and the technicians... The scientific field studied on D1B is getting wider and wider. Beside the classical study of magnetic structures, phase-transformations and surface structures, two experiments on textures were performed in 1988 which proved the exceptionally good performance of D1B in this field. A complete texture study of a polycrystal can be done automatically in less than 2 days, pole figures of high accuracy can then be extracted from the data. The option of polarized neutrons was used to search for ferrimagnetic spin arrangement in Spinel phases and cubic Laves phases. D1B as other instruments did its best to contribute to the knowledge of the new superconductors.

New automatic fitting programs facilitate the data treatment of the kinetic, thermodiffractrometric and texture studies which normally produce huge data files.

## D2B Very-high resolution powder diffractometer on the thermal beam H11

D2B has been very reliable and heavily scheduled in 1988, with average allocations of only 3.5 days. User demand is about 2.5 times available time, which is one of the largest and probably optimal; a larger ratio would mean too many unsatisfied customers. The increasing availability of high resolution powder diffractometers elsewhere has not had any apparent effect on the demand for the ILL instruments.

The machine has been mainly appreciated for the rapidity with which precise structures can be obtained as a function of temperature; programmed temperature control and high flux

have reduced run times to typically one hour, with runs repeated using different detectors to reduce systematic errors. The high resolution mode has been less popular because the samples usually limit the resolution anyway, and sample dependent effects such as strain, particle size, etc. limit the precision of the structure refinement. Of course these sample effects are of interest in themselves, but in most cases are simply a nuisance associated with excessive resolution.

D2B has of course achieved a number of firsts in the study of high temperature superconductors following the earlier successes of D1A; location of oxygen and the co-ordination of Bi in the bismuth cuprates, refinement of three different phases of the thallium cuprates, precise temperature dependence of the low temperature structure of YBaCuO etc. Some of these results could be obtained also on other machines, but the reliability and speed of D2B, and the versatility of its sample environment, have given this machine an apparent advantage.

There are still two main areas for improvement. The monochromator, which gives a shoulder on low angle peaks in the high resolution mode, must eventually be replaced, and a graphite filter is needed to permit routine operation at wavelengths of 2.4 Å and higher. It is now also possible to improve resolution and line shape by inserting a collimator between the monochromator and the sample.

## D3B Polarized neutron diffractometer on the hot-neutron beam H4

D3B, a modernized version of D3, is a single crystal diffractometer designed to map atomic magnetization densities in crystalline compounds down to a subatomic scale with a sensitivity of some milli Bohr-magnetons per $Å^3$. By using the shortest wavelengths available at the ILL, D3B is able to measure magnetic structure factors up to $\text{Sin } \theta/\lambda = 2$ $Å^{-1}$, a value corresponding to a direct space resolution better than any details nature provides in the smallest magnetic ions.

By simply reversing the beam polarization, D3B performs a highly sensitive measurement of the nuclear-magnetic interference amplitude term which is present in the Bragg scattering of polarized neutrons from a small single crystal specimen magnetized in a field.

**Short wavelength polarized neutron beam.** D3B is installed on the hot neutron beam tube H4 and uses readily exchangeable CoFe and Cu2MnAl monochromators. The shaft positioning, biological shield rotation and the carrousel to insert resonant harmonic filters are all motorized so that a wavelength change is an on-line operation, which is particularly useful when extinction or multiple scattering are present. The range of calibration is from 0.42 to 0.84 Å. The highest neutron flux is $10^7$ $cm^{-2}$ $s^{-1}$. The beam polarization is good. It reaches 99% at its best. Polarization reversal is made with a "cryoflipper", it is 100% effective, does not depend on wavelength and is practically insensitive to stray-fields from the sample magnet.

**Magnetic normal-beam diffractometer.** The secondary axis is set on an ILL "Tanzboden" floor, mechanically attached to the monochromator exit port (Fig. 107). Normal-beam geometry with a lifting detector is used: this is most suitable for trouble-free high-field magnetic diffraction work, since it keeps the large cryomagnet vertical. The low-lying air cushion base-plate maximizes the free space under the beam line. The detector support arm and the sample adjustment table are non-magnetic, so cryomagnets with large stray fields can be used. The control of the incident beam size is made with a new motorized, non-magnetic slit system and the pneumatic half-shutter system in front of the 5 cm diameter single $^3$He detector facilitates automatic orientation matrix determination.

**High-field, low-temperature sample environment.** Most measurements carried out on D3B require the sample to be at low temperature and in an intense magnetic field, so the dedicated 4.6 Tesla Oxford Instrument (OI) cryomagnet is almost continuously in use. It is now equipped with an ILL variable temperature insert made fully reliable down to 1.5 K by closed-loop control of the helium feed. An ILL PTC (precision temperature controller) and an OI-CMC (cryomagnet

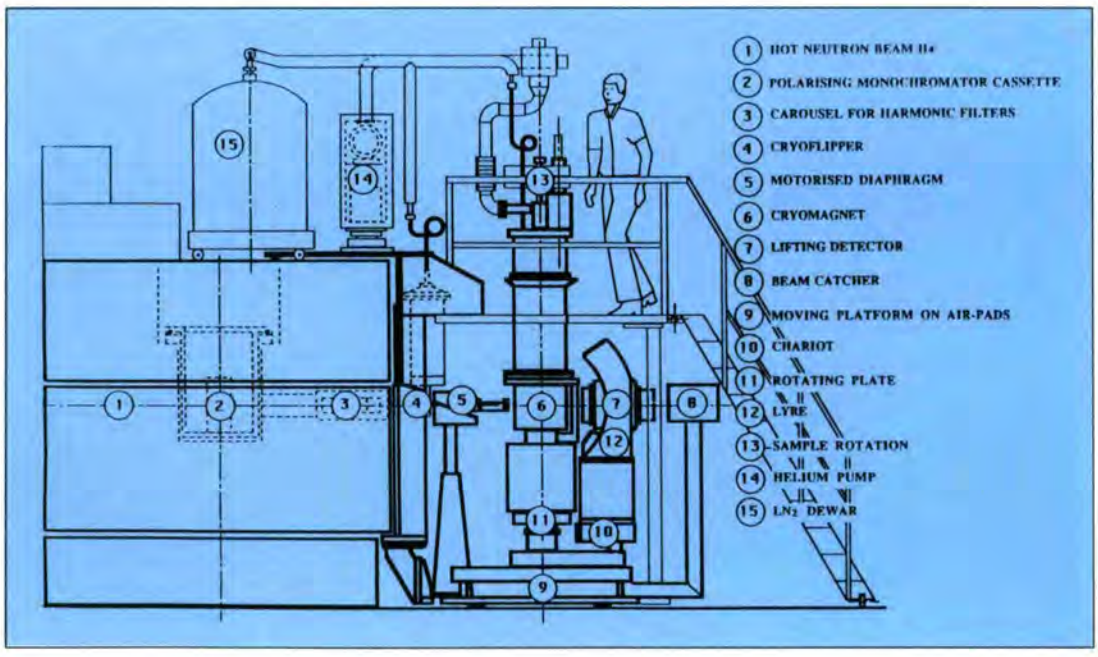



*Figure 107*
*Schematic view of the polarized neutron diffractometer D3B.*

controller) have been interfaced to the instrument computer which can set [H,T] variables on-line in the range 1.5 - 4.62 Tesla, 1.5 - 273.0 Kelvin. An air-tight chamber attached to the top of the cryostat makes it possible to insert dangerous (alpha emitter) or air-sensitive samples without any contact with the atmosphere.

**Data acquisition.** The instrument is extensively automated. Positioning, measurement, data storage and data transfer are under control of a dedicated PDP 11/73 computer. The software system is based on the ILL-MAD acquisition system with an extended version of the original D3 user interface. It provides the user with many commands ranging from elementary actions (such as *SMF 4.62 to set the nominal field, *WAV .843 to select the appropriate wavelength followed by *SBH Er to insert the appropriate Erbium filter) up to sophisticated data collection sequences (like *CBP) which generates a sequence of Bragg reflection indices and measures their polarization ratios.

Without interrupting the data acquisition, the user can prepare in advance Input-stream files containing many such orders by using text editors (EDT, TECO). File connections in routine or sequential modes are straightforward.

## D4B Disordered-materials diffractometer on the hot neutron beam H8

The instrument has been increasingly used at its stability limit of one part per thousand. The VAX computer package (due to A. Barnes) for regrouping the data collected with the small linear multidetectors and for checking the scan reproducibility, detector cell stability, etc. has been very efficiently used. It has allowed us to optimize sample conditioning and sample environment design. At room temperature we could obtain high quality data for the isotopic substitution method. At high temperature (with sapphire single crystal containers) or with pressure cells there is a need to improve the experimental set-up and the data correction procedure.

## D9 Four-circle diffractometer on hot neutron beam H3

D9 has operated very smoothly, taking full advantage of its new operating system for the Micro-VAX II computer. Work on development of environment control units continue, and this year several measurements have been made with a pressure unit going to 25 Kbar. The major new item on D9 is however the new small position-sensitive detector, which covers 32 x 32 pixels of 0.25 x 0.25 deg. Although it still needs some improvement in homogeneity, it has served on several occasions for studies of twinning (Fig. 108) and the limit for satellites. At present work is in progress on data reduction systems, which should make the detector routinely applicable to all kinds of diffraction studies.

## D10 Four-circle triple-axis spectrometer on thermal guide H24

This year D10 has again been scheduled mostly for experiments requiring the versatility of a three axis instrument in combination with an Eulerian cradle. These experiments include the measuring of weak satellite reflections from modulated structures, elastic diffuse scattering from alloys, of Huang scattering and of paramagnetic and magnetic elastic diffuse scattering. Several developments (mainly in the software and sample environment field) made during this year have improved the instrument performance for this type of experiments:

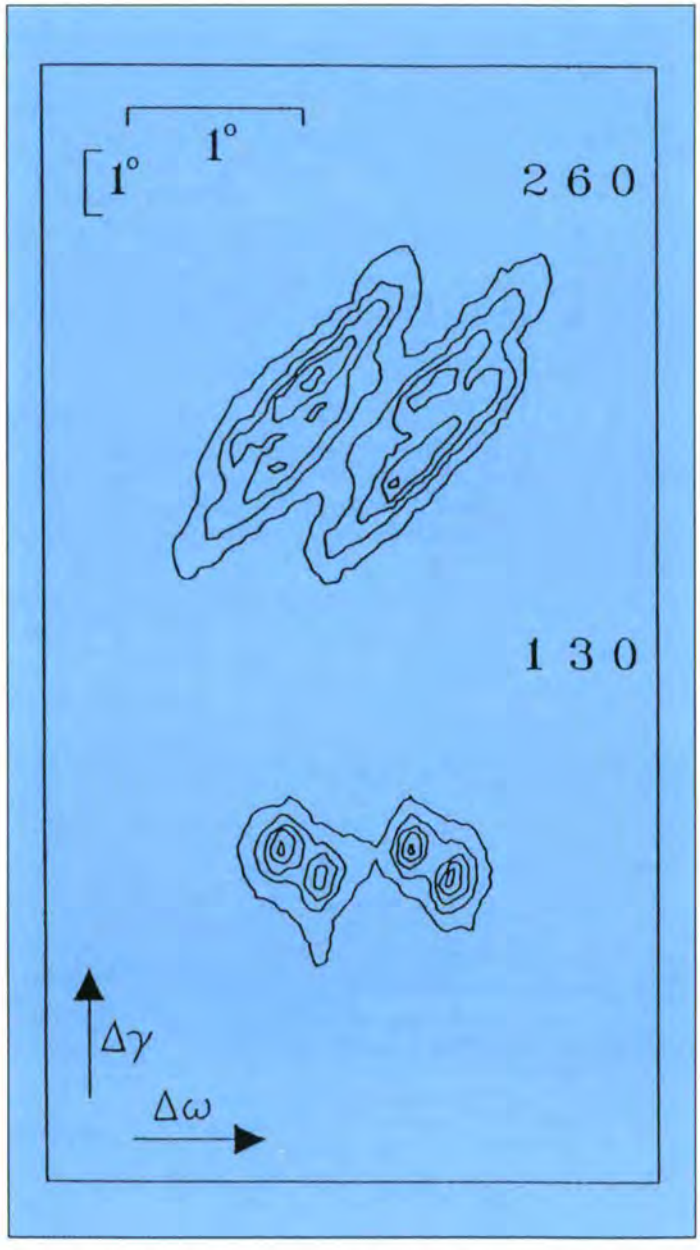



*Figure 108 Measurement of twinned $BaY_2Cu_3O_7$ employing the 2-dimensional PSD of D9. The scan (in ω) is projected onto the ω-γ plane, where γ is the horizontal detector direction. The four peaks due to the twinning are clearly separated.*

The new 4-circle cryostat (S. Pujol, R. Chagnon) is now fully operational. It proved its qualities during several months of successful operation on the instrument. It is now possible to maintain sample temperatures down to 1.7 K, stable within a few hundredth of a degree and independent of the 4-circle setting angles. The helium consumption at these low temperatures is smaller by a factor of 4 than its predecessor at its lowest temperature of 4 K. The cryostat is designed to work as a cryofurnace. A dilution option is under development in collaboration with the CNRS (Benoit).

An orange cryostat equipped with a Helmholtz pair of superconducting coils (Forgan, Gibbons) is now available (belonging to the S21 instrument). This cryomagnet is designed for sample temperatures below 15 K and achieves a maximum magnetic field of 2 T at 2 K.

The long reactor shut-down of the 1988 summer has been used to replace the old PDP 11/34 by a Micro-VAX II as instrument computer. The new hardware allowed the long desired improvement of the instrument software. The operating system (Allibon, Barthélémy, Roure) combines the possibilities of the software for a conventional 4-circle diffractometer with those of a three axis spectrometer. This unique feature allows the user to perform an energy scan (i.e. to find the exact elastic position) and to continue then with conventional 4-circle measurements within the same program. Thus the command syntax is standardized and the parameters for the inelastic work become accessible within the standard parameter menu.

A program DIFFIT (Brückel, Vrtis) for the treatment of data from D10 and some other diffraction instruments (particularly D9 and D15) is now installed on the main computer and will be available on the instrument computers after the next shut-down. The program offers various options to manipulate data (add, subtract, append,...), to fit data with several curve types (including parameter constraints) and to calculate corrections such as a form factor correction for diffuse scattering, a Lorentz factor calculation for Bragg data measured with Q-scans or a Bose factor correction for inelastic scans.

## D15 Four-circle diffractometer on the inclined thermal beam IH4

D15 continues to be used for studies of single crystals under a wide range of external constraints. In the spring an optional extension made to the high level platform allowed the CENG 10 T superconducting magnet to be installed for investigation of the H-T phase diagrams of $U(Pt_xPd_{1-x})_3$ and $FeI_2$. During the long shut-down in the summer the monochromator drum was dismounted to allow the replacement of the H8 beam tube which is underneath.

Thanks to splendid cooperation by all concerned the instrument was reinstalled, realigned and operational within one week of the start-up in August. At the same time the PDP 11-34 control computer was replaced by a Micro-VAX which runs the standard ILL diffractometer control program MAD. Special options are available for normal beam geometry, and precise reflection centering using automated half-shutters to enable crystals mounted in cryostats or furnaces to be accurately positioned on the axis of the diffractometer at the temperature of the experiment.

## D16 Four-circle diffractometer on the cold neutron guide H16

The new monochromator housing has improved the signal to noise ratio on the instrument as well as the incident flux in conditions of high angular resolution. During the year, the PDP-11 computers have been replaced by a Micro-VAX to run data acquisition and analysis programs. D16 is a diffractometer providing information in a range and resolution in Q that lie in between thermal beam diffractometers and small-angle cameras, and a constant effort is made to ensure full software compatibility with these instrument classes. Proposals accepted on D16 in 1988 included experiments on biological membranes, polymer solutions, surface lattices and intercalated compounds, ordering in alloys, magnetic frustration, clays, liquid crystals and micro-emulsions.

## S18 Neutron interferometer and diffractometer on the thermal guide H25

Last year was mainly used to develop the mechanical and electronic set-up for a double crystal interferometer. It consists of a large aluminium box containing the mechanical and electrical installation. Because of its weight the vibration isolation of the optical bench had to be modified. The alignment system now consists of five helical springs. The stretching of three of them is computer controlled.

The crystal movement is realized by stepper motor driven torsion springs. Their resolution is about $2 \times 10^{-5}$ seconds of arc per motor step for the rho axis and $2 \times 10^{-6}$ seconds of arc for the theta axis. The positions of the stepper motors are controlled by encoders which can be read out directly by the PDP11/73 computer. The crystals are cut in a form that allows the simultaneous use of neutrons and X-rays.

Different materials have been tested according to their usability as high velocity phase shifter for future Fizeau experiments.

## D19 Four-circle multidetector diffractometer on the thermal beam H11

D19 experiments in 1988 included crystallographic studies on the A-form of DNA on Vitamin B12 at low temperature on ethylene glycol on the surface of a protein, and on a number of inorganic complexes, as well as phase transition studies and works on liquid crystals in a magnetic field.

Both the banana position-sensitive detector (PSD) and the 15 K Displex cryorefrigerator worked normally. Preparation for replacement of the PDP11 and VAX 11/750 by a Micro-VAX III is well advanced.

It is hoped that in 1989 a start can be made on both a D9/D15–type small PSD with high detection efficiency for wavelengths below 1 Å, and at least one very large 20° x 64° curved PSD. This latter detector could either be of the D19 type, or based on the new D20 detector technology.

## D20 High flux multidetector diffractometer on the thermal beam H11

D20 has worked satisfactorily during his second year of operation with the small position-sensitive detector ($2\theta$ = 12.6°) used in the scanning mode for most of the experiments. A large variety of sample environment has been used: standard Ø = 620 mm vacuum vessel, orange cryostat, furnaces, dilution cryostat, cryomagnet, eulerian cradle, special automatic sample changer for 100 samples, CuBe pressure cell.

Two working areas on the VAX are available for data treatment, D20 and D20L. Both have very similar data regrouping and plotting procedures. D20 is made for powders and joins the D1B data processing, 3 dimensional plots, ABFIT fitting procedure, etc.; D20L is made for liquids and amorphous materials and joins the D4B data processing mode.

November 1988 has been marked by a decisive step in the construction of the large banana (160°) of D20: the glass blades with the microstrip system of A. Oed (32 cells of D20 per blade of 82 x 150 mm$^2$) have been tested successfully. The quality of detection is very good and is the same in the middle of a 32 cells blade as well as at the junction of two blades.

## S20 Neutron topography diffractometer on the thermal guide H24

Neutron diffraction topography is a 'local ' diffraction technique which basically consists of recording on a photographic plate the beam Bragg diffracted by a single crystal. Relevant physical information is obtained by investigating the variations of the intensity within the recorded Bragg spot, and relating them to the inhomogeneities of the sample (defects, domains, coexisting phases,...). A simple diffractometer, S20, which results from the collaboration of the Lab. L. Néel (CNRS) and the ILL, was mainly devoted over the past years to this technique. It was used to produce, for instance, the first observations of several kind of 'exotic' magnetic domains: 180° antiferromagnetic domains in $MnF_2$ and $CoF_2$, chirality domains in the helical phases of Tb, Ho and MnP.

This instrument has been completely renewed in 1988 in order to better perform the kinds of experiments now proposed by the users (mostly investigation of magnetic phase transitions, experimental checks of extinction theories, memory effects of domain structures, "local" investigation of crystal properties...). The complementarity for this type of investigation of neutrons and synchrotron radiation techniques has to be pointed out.

This resulted in both a bigger incoming flux and a smaller background. The instrument can operate at any wavelength within the thermal guide range ($1 < \lambda < 2.6$ Å), and either with unpolarized or polarized neutrons. The passage from unpolarized to polarized work takes only a few minutes because both types of monochromator are present on a "carrousel" within the shielding, and the guide fields and flipper are just attachments which are easily set-up. A big improvement has already been achieved by partial automation of the scans, polarization and temperature (Displex 10-300K), and by a modification which allows recording of Bragg spots which are out of the horizontal plane (±30°).

Special mention should be made of the use of a very high resolution (0.2 mm) position sensitive detector developed at the LAPP, in Annecy. This detector has not only produced on S20 images very comparable to those obtained by photographic methods, but has also allowed original quantitative work (for instance spatial maps of flipping ratios) by simply varying an external parameter and comparing the recorded files. This multidetector is under further development.

## S21 Double crystal spectrometer on the thermal guide H24

This instrument has been used for some new types of experiments this year, including surface investigations and USANS (Ultra Small Angle Neutron Scattering). The latter applications are promising and yield a momentum transfer range beyond $10^{-3}$ Å$^{-1}$ complementary to the D11 camera. Using one channel cut Si (331) crystal and a normal plate at a wavelength of 1.76 Å, Q-values of a few $10^{-5}$ Å$^{-1}$ were obtained from strong scatterers. For longer experiments the time stability of the crystal tilting tables has to be improved.

More fundamental experiments were performed on acceleration and deceleration of neutrons by time dependant magnetic fields. The use of the longitudinal Stern-Gerlach effect or Zeemann splitting has become a standard technique to produce polarized beams.

## DB21 Four-circle diffractometer with PSD for biological macromolecules on the cold guide H15

This instrument is dedicated to single crystal diffraction studies at low resolution with $H_2O/D_2O$ contrast variation, mostly on biological macromolecules. DB21 has been running without major problems almost 100% of the time, except for one break-down of the computer in April. A typical measurement on a crystal of a biological macromolecule lasts one week. The present development of the software MAD by J. Allibon, allows full remote control of the instrument.

The scintillator of the 2-dimensional Position Sensitive Detector (Anger camera) was replaced with success by a new one. A second 2-d P.S.D., a $^3$He gas detector with 1.5 mm resolution and 200 by 200 mm$^2$ area, prepared by J. Jacobé and coworkers is now available. It will be tested in 1989.

Coordinator: C. Zeyen

# Special Instruments and Experiments

## Introduction

1988 was a very successful year in the field of S-activities: probably the biggest experiment ever to be performed at the ILL - the search for neutron-antineutron oscillations - has started the data acquisition. The experiment S50 (precision measurement of $h/m_n$) got its definite apparatus and started measurements. The neutron gravity spectrometer NESSIE with resolutions in the neV range came into operation in November. Construction of two new instruments has been started (the VCN-interferometer on level D and the EVAnescent wave diffractometer EVA for surface studies in the new neutron guide hall); both of them will become operational in 1989. A new project on a sophisticated application of ultracold neutrons for Cavendish's experiment with neutrons is in its design phase.

## Overview of Current S-Activities at ILL

### The current S-activities are listed in the following table

| | | |
|---|---|---|
| S10 | (n, α reactions) | Gent/Mol |
| S18 | Thermal neutron interferometer | Dortmund/Vienna |
| S20 | Neutron topography | CNRS/ILL |
| S21 | High resolution double crystal spectrometer (with an optional γ-beam) | ILL |
| S30 | Concentration profiles by (n,p),(n,α)-reactions | HMI Berlin |
| S34 | γ–γ correlation measurements | ILL/Sussex |
| S42 | Laue diffraction | CNRS |
| S50 | Precision measurement of $h/m_n$ | PTB Braunschweig |
| S51 | Neutron activation analysis | CNRS/ILL |
| GAMS4 | Double flat γ crystal spectrometer | NBS/ILL |
| SN5 | UCN-source on level C for developments | ILL |
| SN7 | Beam of polarized neutrons | ILL |
| H17 | Cold neutron beam | ILL |
| H18 | Cold neutron beam | ILL |
| S-UCN | Superthermal liquid He UCN-source | RAL/HMI Berlin |
| $n\bar{n}$ | Neutron- antineutron oscillations | Heidelberg/Padova/ILL |

### UCN/VCN-activities on level D

| | | |
|---|---|---|
| VCN, UCN-source on TGV + turbine | | TUM/URI/ILL |
| EDM | Search for an electric dipole moment of the neutron | Sussex + international collaboration |
| n-bottle | Neutron lifetime measurement | ILL |
| GD | UCN gravity diffractometer | TUM/URI/ILL |
| NESSIE | UCN gravity spectrometer | TUM/URI/ILL |

### Under construction

| | |
|---|---|
| VCN-interferometer on level D | Vienna/TUM |
| EVAnescent wave diffractometer (cold neutrons) | University of Munich |

### Project

| | |
|---|---|
| Cavendish's experiment with neutrons | International collaboration |

## Progress on Instruments in Routine Operation

Progress on S-instruments in routine operation in 1988 can be summarized as follows (see also the Instrumental Sections of Colleges, especially College III and College Secretaries' reports for scientific results).

**S18** In 1988 the mechanical and electronical set-up for a double crystal interferometer was developed. It consists of a large aluminium box in which the mechanical and electrical installation is housed. Because of its weight the vibration isolation of the optical bench had to be modified. The alignment system consists now of 5 helical springs. Three of them are computer controlled.

The crystals are moved by step motor driven torsion springs. Their resolution is about $2 \times 10^{-5}$ seconds of arc per motor step for the ρ–axis and $2 \times 10^{-6}$ for the θ–axis. Both of these movements are computer controlled with the help of encoders.

The crystals are cut in a form that allows the simultaneous use of neutrons and X-rays. Various materials have been tested on their possible use as high velocity phase shifters for future Fizeau experiments.

**S20** The reconstruction of S20 was finished early this year; the instrument is in routine operation since that time. The collaboration with the LAPP on the development of a 2-dimensional very high resolution position sensitive detector (0.2 mm) is going on.

**S30** In 1988 a new sample vessel was installed that is easier to handle and that shows a strongly reduced background. For each experiment the adequate optimized detector is available. The depth resolution is improved by detector cooling and improved electronics. A facility for coincidence measurements was made available to perform background-free measurements on samples with low impurity concentrations as well as a facility for in-situ annealing up to temperatures of at least 500°C and a facility for low temperature measurements down to -200°C. Further, a rotatable sample holder is incorporated in the sample vessel to perform tomographic examinations of collision cascades after ion implantation and finally data treatment is now performed by a portable IBM-PC with a plug-in Multichannel Analyzer and upgraded software.

The performance of this reconstructed spectrometer is excellent. For example, the signal to noise ratio was improved by one order of magnitude for N, Cl and He analysis.

**S34** The S34 station at the end of the H22 thermal neutron guide is used for $\gamma$–$\gamma$ coincidence and correlation studies. This year a static four detector correlation system was successfully tested. However, hardware restrictions dictate that this facility cannot yet be offered on a regular basis. Additionally the evacuated tube, through which the neutrons reach the sample chamber, was redesigned resulting in a considerable decrease in background from scattered neutrons. With the new arrangement the facility allows also to introduce electron detectors close to the target, thus making possible the study of $\gamma$–$\gamma$ coincidences.

**S50** In 1988 the final apparatus became operational; it was then aligned with neutrons. First measurements of $h/m_n$ show a relative uncertainty of less than $10^{-6}$. Now experiments on the systematic errors will start.

**GAMS4** The double flat crystal spectrometer has been extensively and successfully used for lifetime measurements of excited nuclear states (see blue box, college 3). Great effort was put into a further increase in sensitivity of the instrument. A new Ge-detector with 30% efficiency (measuring the doubly diffracted $\gamma$-rays) has been installed. Due to careful shielding and the installation of additional pre-collimators the background was reduced by a factor $\cong$ 4 for measurements at very small diffraction angles. This is essential for precise measurements of neutron binding energies (which should lead to a redetermination of the fine-structure constant). Important progress was achieved in making the system more user-friendly. The GAMS4 data acquisition system is linked directly to the GAMS2/3 computer using also parts of its on-line data analysis system. It can also be linked to the GAMS1 system installed at the opposite end of the through-tube such that GAMS1 can determine burn-in and/or burn-out of the target material in cases where high neutron cross-sections are present.

**EDM** A preliminary analysis of data collected up to now gives an electric dipole moment of $(-1.2 \pm 0.6) \times 10^{-25}$ ecm. Work is now underway on the development of a much larger storage volume, around 50 l, and the use of a magnometer gas in this volume, along with the neutrons. To this end a deuterated polystyrene surface has been investigated, and found to be promising.

## Progress on new S-instruments and experiments

The **neutron-antineutron oscillation experiment** got its detector installed in 1988 (150 $m^3$ for the detection of the particle shower following the annihilation of antineutrons); the neutron guide on the 2nd cold source out to the detector house (detector axis 154.4 m away from the reactor core) is finished. This experiment started the data acquisition in the last cycle of 1988.

The gravity spectrometer **NESSIE** on the turbine UCN-source was finished in October 1988. Fig. 109 shows the neutron parabolas inside the vacuum vessels. The principle used for the monochromator and the analyzer in NESSIE is range analysis of ultracold neutrons (1 cm $\cong$ 2 neV) that leads to very high energy resolutions. The mode of operation is: variable incident energy and fixed final energy. Flux measurements at the sample position are in excellent agreement with computer simulations. Fig. 110 shows first resolution measurements with a special form of graphite (Papyex) and beryllium oxide as samples. Full width at half maximum values showed to be as low as 16.3 neV. The first real samples will be biological samples (anti-bodies) and model membranes in the first cycle 1989.

Another new instrument, the **VCN interferometer**, is being installed on the through-going VCN beam on level D. This instrument was proposed by a Vienna-Munich collaboration and aims at the application of neutrons with wavelengths between 60 and 100 Å that have distinct advantages over both cold and ultracold neutrons (index of refraction deviates significantly from one; possibility to use standard optical diffraction elements). Research will be directed towards precision tests of the linearity of the Schrödinger equation, of the unitarity of quantum mechanics, topological quantum effects for neutral particles, neutron coherence studies, etc. This long base line interferometer (6 m) will be installed on an optical bench.

The bench in honeycomb technology as well as the pneumatic vibration isolation supports of the bench have been acquired

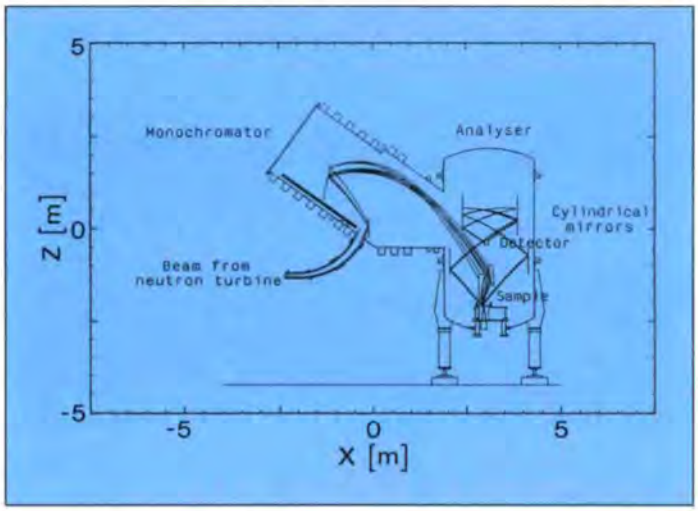



*Figure 109 Vertical section through NESSIE showing neutron paths as calculated by ray tracing.*

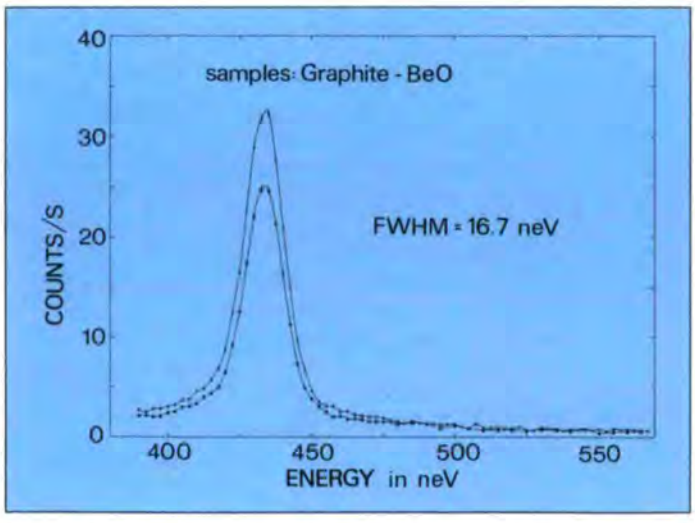



*Figure 110 Resolution measurements of NESSIE on a Papyex (graphite) sample (higher intensity) and a BeO sample showing FWHH of 16.7 neV.*

from the NRC Newport Corporation, tested at the University of Vienna and now installed on the extended platform on level D. First measurements have been performed on the VCN beam as well as first interference measurements on phase gratings that will serve as the optical elements of the interferometer. Sound wave isolation and air-conditioning is foreseen for installation in 1989. This instrument is expected to be fully operational in spring 1989.

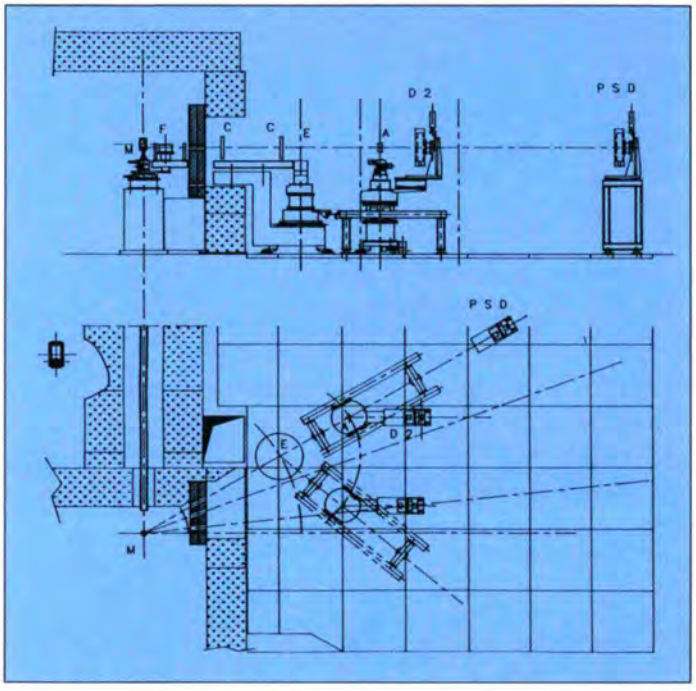



*Figure 111 Vertical and horizontal view of EVA, the evanescent wave diffractometer on H53. M = monochromator, F = filter, C = collimators, E = sample, A = analyzer, PSD and D2 detector positions. This graph has been prepared with the newly acquired CAD system "AUTOCAD" by J.L. Coquin.*

Another S-instrument will become operational in 1989; the **EVAnescent wave diffractometer EVA**. This instrument was proposed by a group from the University of Munich and accepted by the Instrument Subcommittee in 1988. EVA is designed for experiments on surfaces where the scattering and total reflection of neutron waves are combined in order to obtain a surface-sensitive method that retains all the advantages of neutron scattering for the investigation of structural and magnetic properties of solids and liquids. EVA will be placed on a Tanzboden at the new guide H53 and run at a fixed wavelength of 5.5 Å provided by a PG(002) monochromator. The outline of the instrument (Fig. 111) is determined mainly by the necessary collimation of the incident and scattered beam in a plane normal to the sample surface which is typically 1 to 3 mrad for experiments under total reflection conditions.

A 1-dimensional position-sensitive detector from ORDELA with high spatial resolution (≤ 1 mm) and an active area of 200 mm x 50 mm will be used to measure the angular distribution of the scattered neutrons normal to the surface. In order to discriminate against inelastic scattering from the sample an analyzer crystal can be mounted in front of the PSD. The reflection condition is controlled via a conventional $^3$He detector on a vertical translation stage mounted on a table with airpads. The sample surface has to be aligned with respect to the incoming beam such that the total reflection condition is fully preserved when the sample is rotated in order to adjust the correct scattering geometry. For this alignment a stepper motor controlled unit including x-y-z translation stages and two Eulerian cradles have been acquired (Huber). As an "easy-to-modify" instrument, EVA will offer a wide range of possible configurations adaptable to proposed experiments (e.g. liquid samples). The project is well in progress, the site has been prepared and all mechanical and electronical items are scheduled for supply in 1988. The installation is planned for early 1989.

## Project

First ideas about Cavendish's experiment with neutrons and laboratory masses have been discussed; very small deflections of a UCN beam due to tiny forces such as gravity with laboratory masses, electromagnetic fields or may be even the "fifth force" can be sensed in a sophisticated UCN-imaging system of high optical quality. This project is the ultimate challenge in UCN applications for the time being.

Coordinator: W. Drexel

*View of the experimental platform of the "level D" showing the global view of the VCN-UCN experiments.*

# Directorate Services

# Scientific Coordination and Public Relations

## Scientific Programme in 1988

Due to fuel element supply problems the reactor operated for only 5 cycles. There were extended shut-downs in July-August and in December.

Switzerland has joined the ILL.

The beam-time distribution among the member countries is as follows:

*On the occasion of the visit by the French Minister for Research and Higher Education, A. Carignon (Mayor of Grenoble and French Minister for the Environment, left) and B. Maier (ILL Scientific Secretary, right) exchanged views on ecological problems.*

## Beam-time allocation in 1988

**(subcommittees October 1987 and April 1988)**

| Country | | all instruments | | all instruments except nuclear physics and scheduled special beam instruments | |
|---|---|---|---|---|---|
| | | in days | in % | in days | in % |
| F | only | 533 | 9.3 | 374 | 6.4 |
| D | only | 754 | 13.1 | 503 | 8.7 |
| GB | only | 866 | 15.0 | 794 | 13.8 |
| E | only | 50 | 0.9 | 50 | 0.9 |
| CH | only | 17 | 0.3 | 17 | 0.3 |
| F | in collaboration | 612 | 10.6 | 522 | 9.1 |
| D | in collaboration | 1041 | 18.1 | 627 | 10.9 |
| GB | in collaboration | 719 | 12.5 | 484 | 8.4 |
| E | in collaboration | 88 | 1.5 | 74 | 1.3 |
| CH | in collaboration | 40 | 0.7 | 40 | 0.7 |
| **overall total** | | **5753** | **100.0** | **3834** | **66.6** |

# Instrument Statistics 1988

| Instrument | Beam-time requested (days) | Beam-time available (days) | College 3 | College 4 | College 5 | College 6 | College 8 | College 9 | Industry |
|---|---|---|---|---|---|---|---|---|---|
| D1A | 95 | 67 | | | 60 | | | | 7 |
| D1B | 300 | 173 | | | 121 | 13 | | 39 | |
| D2B | 412 | 173 | | | 165 | 7 | | | 1 |
| D3B | 295 | 154 | | | 154 | | | | |
| D4 | 258 | 102 | | | 10 | 92 | | | |
| D7 | 192 | 158 | | | 112 | 24 | | 6 | |
| D9 | 402 | 177 | | | 117 | | | | |
| D10 | 227 | 166 | | 5 | 110 | 51 | | | |
| D11 | 382 | 192 | | 1 | 15 | 17 | 82 | 74 | 3 |
| D15 | 297 | 170 | | | 170 | | | | |
| D16 | 307 | 188 | | | 19 | 12 | 66 | 91 | |
| D17 | 293 | 185 | | 8 | 28 | 25 | 15 | 108 | 1 |
| D19 | 239 | 176 | | | 86 | | 83 | 7 | |
| D20 | 120 | 56 | | | 27 | 25 | | 4 | |
| DB21 | 133 | 208 | | | | | 125 | 7 | |
| IN1(3-axis) | 233 | 65 | | 55 | | 10 | | | |
| IN1BeF | 54 | 23 | | | | 10 | | 13 | |
| IN3 | 106 | 80 | | 42 | | 17 | | 21 | |
| IN4 | 234 | 160 | | 123 | | 12 | | 25 | |
| IN5 | 378 | 173 | | 15 | | 48 | 10 | 100 | |
| IN6 | 370 | 169 | | 46 | | 79 | 8 | 36 | |
| IN8 | 481 | 166 | | 142 | | 20 | | 4 | |
| IN10 | 339 | 166 | | 8 | | 56 | 5 | 97 | |
| IN11 | 495 | 176 | | 46 | | 27 | 6 | 97 | |
| IN12 | 362 | 143 | | 119 | | 24 | | | |
| IN13 | 265 | 166 | | 7 | | 31 | 7 | 121 | |
| IN20 | 347 | 168 | | 72 | 96 | | | | |
| PN1 | 283 | 208 | 208 | | | | | | |
| PN2 | 355 | 187 | 187 | | | | | | |
| PN3/4 | 548 | 195 | 195 | | | | | | |
| PN8 | 352 | 176 | 176 | | | | | | |
| **Total** | **9164** | **4765** | **766** | **689** | **1350** | **600** | **407** | **850** | **12** |
| | | | 16% | 14% | 28% | 13% | 8% | 18% | |

College 3: fundamental and nuclear physics

College 4: structural and magnetic excitations

College 5: crystallography and magnetic structures

College 6: liquids, disordered materials and metal physics

College 8: biology

College 9: chemistry, small and large molecules, colloids, polymers

This table represents the beam-time allocation (240 days) of the Scientific Councils of October 1987 and April 1988. Due to fuel element supply problems only 5 cycles were run instead of 5 1/2. Therefore, only 220 days with the reactor at full power were available. The 20 days (half a cycle) allocated in April 1988 will be made up in the first cycle in 1989.

### Some comments to the table

The powder diffraction instrument, D1A, is back to scheduling (2/3 of the beam-time available) since the Scientific Council of April 1988. The new single-crystal diffraction instrument, D3B, (polarized neutrons) was moved to H4 and was commissioned in the first third of 1988. D4, IN1(three-axis) and IN1BeF share the beam-time on H8. Due to the break-down of the D17 multidetector, this small-angle spectrometer was not available in the 5th cycle. The date of the restart of D17 in 1989 is not yet fixed. The three-axis spectrometer IN3 was only available for 50% of the time normally scheduled, and with the commissioning of IN14 at the beginning of 1989, IN3 will only be available for special-beam allocations. IN5, the time-of-flight spectrometer, worked satisfactorily by using a spare D11-detector arrangement for special experiments. The backscattering spectrometer IN10 operated successfully with the application of the new VME electronics.

The special-beam experiments are not included in the present table. They are fully described in the section "Instrument Operation Department" of this report.

## Workshops and Public Relations

Two Workshops, on Quasicrystalline Materials (21-26 March 1988) and on Dynamics of Disordered Materials (26-28 September 1988) were organized by SCAPRO.

SCAPRO also had the responsibility for the local arrangements of the International Conference on Neutron Scattering with about 450 participants, which was jointly organized with the CEN-Grenoble.

The Office has edited a new version of the description of Neutron Research Facilities at the ILL (January 89 edition), the Yellow Book, which is available on request. A very up-to-date desk top publishing system has been applied which is expected to considerably facilitate and enhance the production of future versions of the brochure.

# Library

## Library Budget 1988: 720 KF

In 1988, the Library continued to provide users (I.L.L. and E.S.R.F.) with information and scientific literature. It continued to have a heavy work load despite the part-time secretarial help obtained from the E.S.R.F.:

- 350 purchase orders were typed and processed (250 for the I.L.L. and 100 for the E.S.R.F.).
- 670 books were processed:
- 520 books for the I.L.L. (of which 140 deposited with departments).
- 150 books for the E.S.R.F.
- 650 volumes of journals were bound.

The number of journal subscriptions remained steady (400 of which about 40 for the E.S.R.F.).

In closer connection with the scientific work of the I.L.L.,

- 400 I.L.L. publications
- 500 I.L.L. experimental reports

were listed and put into the computerized data base of I.L.L. publications and experimental reports.

The number of computerized bibliographic searches continued to increase.

The acquisition of a Macintosh was made in order to begin as soon as possible computerization of holdings thanks to existing "user-friendly" software (Hypercard): connection to the cluster and recent agreements between Apple and DEC are the guarantee of easy communication and wide access of users to future library catalogues.

# Safety, Medical and Health Physics Group (SPS)

The units responsible for General Safety, Radiation Protection and the Works Medical Service have as their essential function to support the ILL departments and the guest scientists, to cover the risks associated with the various activities at the ILL.

## General Safety and Radiation Protection

In addition to the everyday work of advice, monitoring and inspection referred to above, these units have carried out or assisted with the following work:

- Continuation with the renovation of fire protection equipment.
- Preparation of safety instructions: use of solvents, specific problems, distribution and use of neutron beams, etc.
- Preparation of safety documents on the use of highly active sources and the $n\bar{n}$-experiment.
- Calculations for shielding around the neutron guides.
- Tests on neutron guides from the horizontal cold source.
- Commissioning of the new station dealing with waste and monitoring of low activity liquid effluents; decontamination and dismantling of the previous station.
- Study for cutting up, processing and evacuation of high activity solid waste.
- Introduction of computerized calculation methods for evaluation of the radiological consequences of an accident.

## Works Medical Service

This service acts as an adviser to the Director, and also provides clinical, biological and radio-toxicological monitoring for ILL staff and scientists working here. It has its own facilities for investigation, and makes use of external organisations for certain tests (ophthalmology, radiology, radio-toxicology).

It collaborates closely with the General Safety and the Radiation Protection units to define the working conditions and evaluate the risks, to keep them at a level at which they have no effect on health.

It also provides the necessary medical attention, and vaccinations, and informs and advises staff on the basis of medical findings and of their requests.

# Instruments and Methods Department

# Instruments and Methods Department

## Introduction

Le DIM est chargé de la réalisation et de la maintenance des instruments (hors informatique) mais aussi du support technique au développement des méthodes de mesures avec neutrons. Parmi les réalisations en 1988 dans le domaine des instruments on peut citer :

- des progrès importants pour les machines tournantes grâce à la réalisation pour D17 d'un nouveau sélecteur, et à la mise au point de nouvelles roues de choppers en matériaux composites capables de vitesse périphérique de 640 m/s ;
- le début d'exploitation sur IN10A d'un système de contrôle et d'acquisition réalisé en VME ;
- les réglages, tests et mise en exploitation provisoire des faisceaux de neutrons issus de la nouvelle source froide horizontale ;
- le début de l'exploitation du nouveau 3-axes IN14 ;
- la fabrication ou le lancement de la plupart des composants des nouveaux instruments IN10C, IN15, D22.

Mais cette année marque aussi le début d'un certain transfert des besoins vers le développement des méthodes de mesures (monochromateurs, détecteurs, supermiroirs) à la satisfaction desquelles l'ensemble du département est appelé à contribuer. C'est dans ce cadre qu'il faut évoquer :

- la signature avec la CERCA d'un contrat de collaboration selon lequel, et moyennant redevance, cette firme fabriquera l'ensemble des multidétecteurs commandés tant par l'ILL que par les laboratoires extérieurs ;
- la poursuite des travaux de réalisation du détecteur banane destiné à D20 et pour lesquels des résultats décisifs sont attendus en 1989 ;
- l'installation d'une source $\gamma$ sur le four de pressage de Graphite pyrolitique qui, en permettant le contrôle continu de la mosaïcité en cours de traitement, fait de cette installation un outil unique et performant en vue de la production par l'ILL des cristaux mosaïques.

Enfin, l'année 1988 est aussi la première année d'exploitation :

- d'un poste de travail en CAO (EUCLID),
- d'un logiciel de calculs aux éléments finis,
- d'un poste de tracé de schémas sur Macintosh en vue de la réalisation de notices d'exploitation.

## Introduction

The Instruments and Methods Department (DIM) is responsible for the construction and maintenance of instruments (apart from computers) and for technical support for the development of measuring methods with neutrons. Work in 1988 in the field of instruments included the following:

- considerable progress on choppers with the completion of a new velocity selector for D17, and the development of new chopper discs of composite materials capable of peripheral speeds of 640 m/s;
- the start of operation on IN10A of a VME control and acquisition system;
- adjustments, tests and preliminary operation of neutron beams from the new Horizontal Cold Source;
- start of operation of the new 3-axis instrument IN14;
- initiation or completion of manufacture of the majority of the components of the new instruments IN10C, IN15, D22.

This year is also characterized by the initiation of a move towards the development of measuring methods (monochromators, detectors, supermirrors) to which the whole of the Department is required to contribute. In this connection should be mentioned:

- the signature with CERCA of a collaboration contract under which CERCA will manufacture under licence all the multidetectors ordered by ILL and by external laboratories;
- continuation of work on the banana detector for D20, for which important results are expected in 1989;
- installation of a $\gamma$ source on the pyrolitic graphite hot press which, by permitting an "on-line" check of the mosaicity during the process, makes this a powerful, unique tool for the ILL production of mosaic crystals.

Finally, 1988 is also the first year of operation of:

- a CAD (EUCLID) workstation,
- finite element calculation software,
- a diagram facility on Macintosh for use in the production of operating instructions.

## Einleitung

Die Abteilung Instrumente und Methoden (DIM) trägt die Verantwortung für die Realisierung und Instandsetzung der Instrumente (mit Ausnahme der Informatik) sowie für die technische Unterstützung bei Entwicklungen von Messmethoden mit Neutronen. Folgende Realisierungen im Jahre 1988 im Instrumentenbereich sollten erwähnt werden:

- wesentliche Fortschritte bei den Geschwindigkeitsselektoren durch die Realisierung eines neuen Selektors für D17 und die Entwicklung neuer Chopper-Räder aus Verbundwerkstoff, die eine Umfangsgeschwindigkeit von 640 m/s ermöglichen;
- Inbetriebnahme eines neuen Kontroll- und Datenerfassungssystems auf IN10A in VME Standard;
- Justierung, Test und provisorische Inbetriebnahme der Neutronenleiter der neuen horizontalen kalten Quelle;
- Inbetriebnahme des neuen Dreiachsen-Spektrometers IN14;
- Herstellung oder Auftragsvergabe der meisten Komponenten der neuen Instrumente IN10C, IN15, D22.

Dieses Jahr kennzeichnet aber auch den Beginn einer Schwerpunktbildung bei der Entwicklung von Messmethoden (Monochromatoren, Detektoren, Superspiegel), wozu die gesamte Abteilung beiträgt. In diesem Rahmen müssen erwähnt werden:

- die Unterzeichnung eines Lizenzvertrages mit CERCA, wonach diese Firma sämtliche sowohl vom ILL als auch von externen Forschungszentren bestellte Multidetektoren herstellen wird;
- das Fortführen der Arbeiten an dem für D20 bestimmten Bananendetektor, deren entscheidende Ergebnisse 1989 erwartet werden;
- der Aufbau einer $\gamma$-Quelle am Pressofen für pyrolitischen Graphit, die die "on•line" Kontrolle der erreichten Mosaizität ermöglicht und so aus diesem Instrument ein einmaliges und im Hinblick auf eine Produktion von Mosaikkristallen am ILL leistungsfähiges Werkzeug macht.

Schliesslich ist das Jahr 1988 auch das erste Betriebsjahr für

- einen Arbeitsplatz für rechnerunterstütztes Zeichnen (EUCLID),
- eine Software für Berechnungen nach der Methode der finiten Elemente,
- einen Arbeitsplatz zur Erstellung von schematischen Darstellungen für Bedienungsanleitungen auf Macintosh.

# Project Office

## Financial Monitoring of Projects

The financial management of instrumentation projects has just been set up on a Macintosh II on a 4D data base linked to the Administration Department's MISCOM computing system. The previous program on PDP11 had in fact become unusable since the end of the contract with CISI in January 1987. The implementation of the new system has been sub-contracted to the companies DEAL and SIVEA. We hope that this will be operational at the beginning of 1989.

## Layout of Instrument Sites

The arrangement of instruments and layout of their sites are now effected by CAD. A workstation using AUTOCAD software has been operational since September. The overall layout plan for the new guide hall ILL22 and the first layout of a new instrument have already been done with this system.

## Structural Design by the Finite Element Method

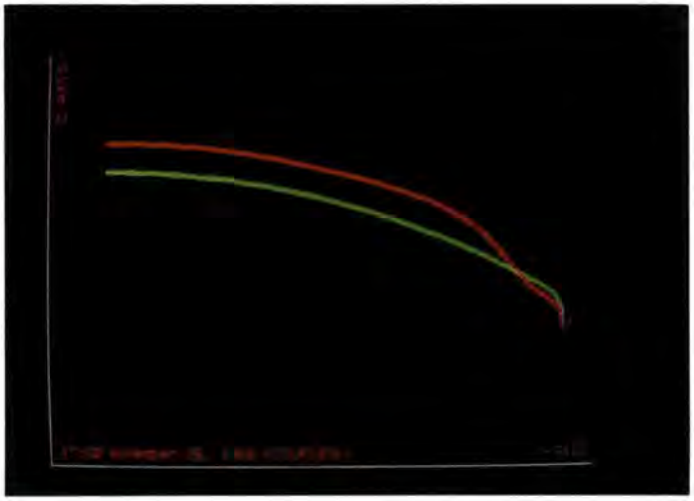
After a training period on the finite element method and its application to linear elasticity and thermo-elasticity, we have been able to use the 'CADSAP' structural design software. The problems covered include:

- dimensioning of the curved surfaces of the large gas multidetectors subjected to internal pressure (HMI, D22, Saclay),
- design of sample holder for high pressure experiments (5000 bar) (see photo).

The optimisation of the thickness of the material through which the neutrons have to pass inevitably requires the elastic limit of the material to be exceeded locally and in a limited part of its thickness. This can only be done with a thorough knowledge of the plastic state in general, the plastic behaviour of the material used and of the possible failure mode to which the structure is liable. Work is being done in this direction.

# Mechanical Construction

The group has continued its support for the scientists as regards maintenance, improvement of experimental equipment, and design and installation of new instruments, despite a considerable reduction in staff.



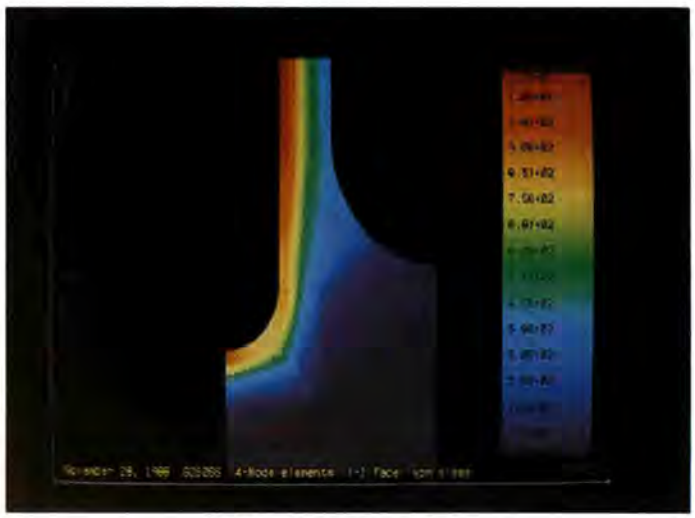


*Illustration of the use of "CADSAP" (Finite element linear elasticity structure calculations), a program for the determination of displacement, deformation and strain fields.*

*The upper photograph shows a section of the torus shaped aluminium bottom of the large multidetector prototype of D22 (Øext. = 1700 mm) exposed to an internal pressure of 2.5 bar. The non-deformed structure is green, the deformed structure is red (the displacements z are scaled by a factor of 20).*
*The lower photograph is a section of an axially symmetrical high pressure cell of ZrTi alloy exposed to an internal pressure of 5000 bar (slimmed cylinder* $Ø_{int.} = 6$ *mm,* $Ø_{ext.} = 12$ *mm).*

## Drawing Office

Priority was given to design studies of equipment for IN14, which has just started operation; also on the primary equipment for the H5 beam-tubes to permit the experiment to start and on the construction of a new completely non-magnetic collimator on D11.

A number of design studies for work to be carried out in future years were only possible with external assistance. The following may be mentioned:

- modifications to instruments D3, T13, IN4
- design studies for the new instruments IN15, IN10, D22
- the definition of the vessels for various multidetectors.

These studies sub-contracted out represented the equivalent of 1.5 additional staff each month.

On equipment the following points should be noted:

- the establishment of a workstation for producing diagrams on Macintosh, one of the uses of which has been the preparation of operating instructions for the instruments D11 and IN14, and the electronics associated with the multidetectors;
- the initiation of a real CAD activity with EUCLID software. To limit the initial investment while confirming that the correct choice had been made, the first jobs were carried out with a single workstation shared between two drawing offices. As regards DIM, one draughtsman was specifically allocated to this task. After an introductory training course, he was able to start on a number of projects for detector vessels (cf design of the D20 'banana') or the instrument D22.

In addition the archiving of manufacturing drawings for Franke & Heydrich standard modules was carried out by a Technical University student.

It is too soon to draw conclusions after less than one year of half-time operation. It must however be recalled that ESRF is operating three (and soon six) CAD work stations, after recruiting qualified and experienced staff. The users and system managers have started an exchange of experience on problems encountered.

## Workshop and Mechanical Construction

In 1988 the activity in the Workshop was particularly intensive, despite a reduction in staff, reducing to four the number of staff capable of operating the six machines.

231 jobs representing 4761 hours of work were dealt with, of which

62% (2970 hours) were contracted out; (a further 10,000 hours of work were also sub-contracted to external firms).

38% were direct requests to the Head of the Workshop:

- by scientists or instrument technicians - 790 hours or 17% of the total,
- by the Reactor Department - 639 hours or 13% of the total,
- for adjustment work during assembly - 362 hours or 18% of the total.

It is interesting to note that:

- most of the jobs given directly to the Workshop were for machining of less than 10 hours;
- the average duration of the jobs sub-contracted out was of the order of 50 hours.

These figures show clearly the important role of the ILL Workshop to ensure a fast response time.

The Workshop built a number of mechanical assemblies which could not be sub-contracted because of the close monitoring required by the users. It contributed to the development of beam-shutters inside the beam-tubes, the construction of mechanized sample-holder assemblies, air film systems such as IN10 and test facilities for selectors and choppers.

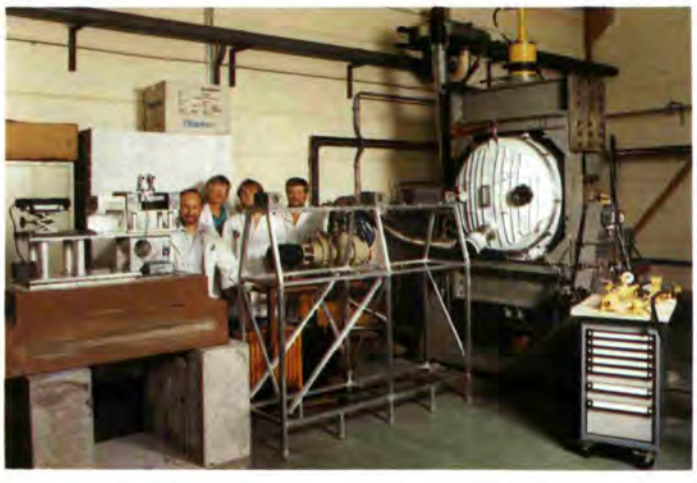
Finally, in cooperation with industry:

- the construction of chopper discs made of composite materials which, despite the embrittlement due to the neutron beam apertures, attain speeds of 24000 rpm, corresponding to 640 m/s peripheral velocity;
- the construction of a selector for D17 made of composite materials; equipped with 140 helicoidal plates, this provides at 5000 rpm a resolution of 10% for 8 Å. This solution was adopted for the future IN11 selector because of its excellent flexibility of application and the complete non-magnetism of its components.

## Test Workshop

The Test Workshop group was particularly involved with the assembly of neutron guides associated with the new Horizontal Cold Source, the installation of IN14 and its automated equipment, and the setting-up of the IN10 monchromator and the first elements of IN15. In addition assistance was given with:

- measurements of mechanical constraints and of deformation of multidetector membranes;
- the development and operation of the production furnace for graphite mosaic crystals (see photo);
- the implementation in collaboration with the monochromator group of a precision glass cutting machine, capable of cutting glass sheets of a thickness 0.5 to 1 mm with an accuracy better than 20 µ over 20 cm length.



*Furnace for drawing the pyrolytic graphite monochromator crystals.*

# Electronics Group

This group is responsible for all construction and maintenance work on control and data acquisition electronics on instruments used at ILL under the scientific programme or within the Department.

During 1988 we saw the first results of several years' efforts both to standardise the electronic modules used on different instruments as far as possible, and to develop on the basis of the VME Bus, new and more powerful modules which will equip the majority of the new instruments before progressively replacing CAMAC.

1. The instrument IN10A which was adopted as the prototype VME-controlled instrument was commissioned during the year. Data acquisition was controlled by a new time-of-flight unit adapted to the Doppler machine.

2. The time-of-flight unit prototypes (adapted to choppers, or designed to control the transmission and phasing of velocity selectors) were also tested during the year. They make it possible to envisage in 1989 the start of the replacement of the time-of-flight instrument controls on IN5, IN6 and IN4.

3. Finally the new instrument T13A for testing monochromators is VME controlled.

The first ASIC circuits were produced with the aid of external laboratories.

The group was also concerned with the construction and start-up of new instruments:

- D3B transferred to a hot neutron beam,
- IN14 first instrument using the new Horizontal Cold Source,
- CAMAC control of the graphite hot press,
- Renewal of the D16 acquisition system,
- Construction and commissioning of the new instrumentation for IN12.

*Final tests and alignment of the monochromator assembly for IN10C in collaboration with the mechanics workshop.*

# Monochromators

T13A: Two-axis neutron diffractometer (under reconstruction)

T13C: Two-axis neutron diffractometer on the thermal guide H23

Li2A: X-ray triple-axis diffractometer (under reconstruction)

Li2B: X-ray orientation unit

Li3A-F: $\gamma$-ray diffractometers

Graphite facility with on line $\gamma$-ray monitoring

Laboratory for crystal preparation.

The departures of A. Freund and R. Hustache to the ESRF necessitated changes in the staff. The group is now headed by A. Magerl. P. Florès will join this group by the end of the year. Another technician, A. Escoffier, has been on detachment for 6 months within the framework of a joint venture between the ILL monochromator group and the ESRF optics group.

In spite of the reduced availability of staff, the group had to cope with a continuing high demand for manufacturing, controlling and improving crystal monochromators, mainly for the ILL and to a lesser extent also for other institutes. In this context the monochromators and analyzers for IN14 and IN10C (see photograph), two new instruments on the 2nd cold source, and D9B have been prepared. The group is also constantly asked to provide polarising $Cu_2MnAl$ Heusler crystals. However, these needs can only be satisfied at a slow pace in view of the available resources.

During 1988 the workload for the group related to "major projects" has increased. In particular, the development of Be as a monochromator material has to be mentioned here. The work can be divided into two parts:

i) the growth of single crystals. This is carried out at the MPI in Stuttgart and crystals with a small intrinsic mosaicity of a few minutes of arc and with a diamter of 15 mm and a useful length of about 50 mm can be produced reliably. However, the present rate is very slow and only 12 crystals have been made this year, which is less than needed to make one monochromator. Obviously, a considerable effort has to be made to solve this production problem.

ii) plastic deformation of the Be-crystals to achieve a mosaicity of 20' to 40'. This is a difficult task in view of the pronounced anisotropic behaviour for plastic deformation of Be, its brittleness, its tendency to twin formation and, last but not least, its toxicity. Nevertheless, it has been shown in the past that some deformation modes exist which, in principle, give the desired mosaic spread. However, to date no crystal suitable for a neutron monochromator is available and consequently this part of the Be-project still has to be considered as a research problem. The ILL in close collaboration with the MPI Stuttgart and the TH Aachen has become engaged in an active programme to investigate various possibilities to increase the mosaicity of the crystals. Spark erosion has been one line of

attack and a systematic study has been performed. Unfortunately, it turned out that this technique is not suitable because of both the limited depth which is perturbed and an unfavourable diffraction profile of the deformed part of the crystal showing long tails. Present efforts concentrate on plastic deformation above room temperature, and we expect to have decisive results beginning 1989.

Significant progress has been achieved in the SiGe project and, in particular, for the preparation of crystals with a fixed Ge concentration up to about 10 atomic %. A monochromator suitable for IN10 has been assembled and a first result is shown in Fig. 112. Problems have been encountered at higher Ge concentrations. These alloys are epitaxially grown onto a Si substrate at high temperature. On cooling they become strongly curved due to the different thermal expansion coefficients. However, it was demonstrated by a complex transport technique that the Si substrate can be removed gradually from one side while the alloy is deposited on the other side. At the end, there is only a flat SiGe crystal left. It can be expected that crystals for monochromators with further increased Ge concentration will be produced during 1989.

Concerning the graphite project, the hot press was moved from the CNRS to the ILL (Hall d'Essais) and the g-ray source was installed in July 1988. This makes it possible to measure the mosaic spread on-line with satisfactory signal to noise ratio and reasonable acquisition time. With this new tool it is possible to stop the treatment at high temperature when the mosaic spread is minimal. A high efficiency is ensured in this way. Furthermore new lines of research have been identified which should permit the mosaic spread to be lowered in the future. So far 0.6° on large plates and 0.4° on small samples have been achieved.

## Status of the Instruments

T13A: It has been agreed to build T13A at the end position of the guide H23. The construction of the primary shielding and of the mechanics for the monochromator changers are under way. The Tanzboden floor will be contiguous with the area of T13C. This allows an easy change of equipment between these two test instruments. The electronic equipment will be VME based.

T13C: continues to be in great demand for monochromator testing and assembly. This diffractometer is also much appreciated by the ILL user community as a versatile diffractometer with quick access to perform test experiments or crystal characterization prior to scheduled experiments.

Li2A: Major reconstruction of the mechanics, electronics and software is being done on this triple-axis X-ray machine. It is scheduled to become available in spring next year.

Li2B: is mainly used for crystal orientation by Laue diffraction either by the monochromator group or by users in self-service mode.

Li3A-F: As last year, only the Cs source has been available due to the temporary shutdown of the Siloë reactor. The Au source will become available again beginning 1989. This will provide the increased flexibility and performance of this installation which is vital for the characterization of monochromator crystals.

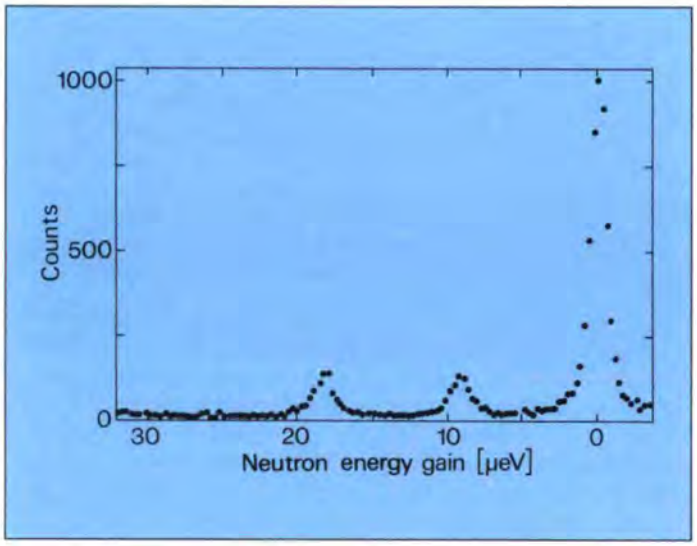



*Figure 112 Tunnel spectrum of $(NH_4)_2\,IrCl_6$ measured on IN10 with a new $Si_{0.9}Ge_{0.1}$ monochromator. Note that the dynamical range has been doubled compared to the usual Si monochromator without deteriorating the energy resolution.*

*Set-up for the measurement of higher order contamination after the second deflector of IN10C during a test phase in the summer (1988).*

# Multidetector Group

The construction of multidetectors and development of the associated electronics is a field in which external and internal requirements have increased so much in recent years that it is difficult to cover them with the limited facilities of the group.

ILL has signed a licence agreement with an external firm. This company will build all the detectors ordered by ILL or by an external laboratory, provided that the detectors are defined and prototypes exist. This type of assistance will be extremely useful and should enable ILL to devote itself exclusively to development and maintenance problems.

## Major Work Carried Out During 1988

- Design and development of delay line electronics for D9,D15 and the detectors under construction for ORPHEE (Saclay).
- Development of a data acquisition system based on a compatible PC to permit tests of detectors.
- Complete renewal of the cabling and of the grouping principle for the 1450 detectors of IN5.
- Construction of IN10C 28 detector bank.
- Construction and initial qualification tests for the IN15 multidetector (32 x 32 cells at 10 mm intervals).
- Construction of a special monitor for IN10C.
- Development and tests of a replacement detector-electronics assembly for D11.
- Installation on D9 of a multidetector of 32 x 32 cells at 2 mm intervals.
- Construction of new vacuum filler equipment for detectors.
- Various work on D13C, D15, DB21.
- With external financial support the group gave assistance:

- to ORPHEE (Saclay) for acceptance inspection of the electronics for Banana type detectors with 400 and 640 cells
- and for the implementation of a provisional detector of 128 x 128 cells at 1.5 mm intervals
- to HMI (Berlin) for the supply of electronics for 400 detectors for a TOF instrument and for the construction, assembly and initial tests of a large detector of 64 x 64 cells at 15 mm intervals, which will serve as a prototype for D22.

## Banana Detector for D20

The considerable efforts involved in the construction of a 1600 cell 160° banana for D20 must also be mentioned.

All the components of this detector now exist and decisive advances were made in 1988 in the construction of electrodes using the photolithography technique to produce metallic microstrips on a glass substrate as proposed by A. Oed.

A positive result was achieved in a test in November on a prototype comprising 96 cells deposited on 3 adjacent glass sheets [1]. This fundamental test made it possible:

- to confirm the validity of the process and in particular of joints between adjacent plates (Fig. 113);
- to prepare the final technical specifications for production of the series of 50 plates necessary;
- to confirm the choice for amplification and localisation of electronic modules already standardised at ILL.

*[1] cf test report on the prototype D20 by A. Oed, P. Convert, M. Berneron, H. Junk - in the press.*

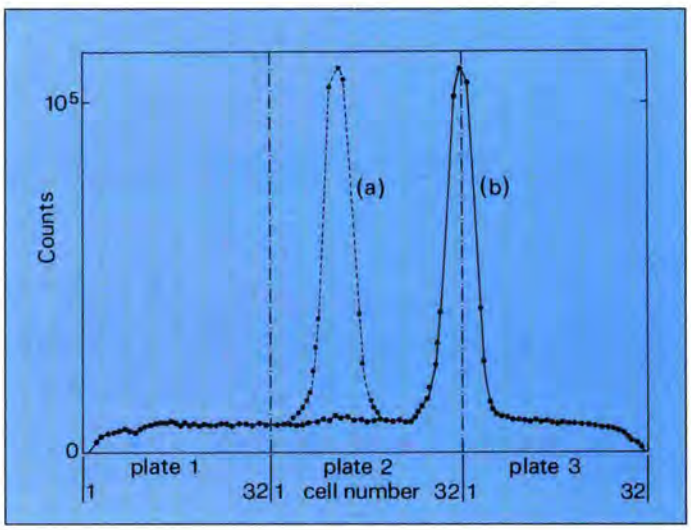



*Figure 113 Neutron Bragg reflection of iron powder measured with the prototype detector of D20 which consists of three micro-strip plates.*

# Multilayer Laboratory

The two evaporators operated with excellent reliability. A larger proportion of the time than last year (32%) was devoted to the development of supermirrors with improved performance. As a result only an area of 17 $m^2$ of titanium-cobalt supermirrors was produced for D7 during the last twelve months.

In collaboration with RIBBER and CNET, the layers of a supermirror were analyzed by AUGER spectrometry. The results, partly published in [2], showed the necessity to modify the production process.

The technique for increasing the divergence of the supermirrors was applied for deposits on the focussing guide produced-by CILAS-ALCATEL for IN10C and in part for the guide elements produced by NTK for KFA and GKSS.

All the work for external laboratories (KFA Jülich, GKSS Geesthacht and IRI Delft) did not exceed 3% of the time. The income from the work done in 1988 permitted the replacement of the MICRO 1 computers on the two evaporators by 11/23+ systems with Winchester discs.

The production of microguides (started at ILL by evaporation of aluminium and nickel) for 450 mm titanium and 50 mm nickel multilayers by sputtering was sub-contracted to BALZERS. Measurements with electron microscope and with neutrons on S3B are in progress.

## Preprints:

*[1] M. Rossbach, O. Schärpf, W. Kaiser, W. Graf, A. Schirmer, W. Faber, J. Duppich, R. Zeisler: The use of focussing supermirror neutron guides to enhance cold neutron flux rates.*

*[2] O. Schärpf: Comparison of theoretical and experimental behaviour of supermirrors and discussion of limitations.*

*[3] O. Schärpf: Properties of beam-bender type neutron polarisers using supermirrors.*

# Reactor Operation Department

# Reactor Operation Department

## Introduction

Cette année, le programme de fonctionnement du Réacteur a dû être établi en tenant compte des difficultés d'approvisionnement en combustible. Deux arrêts relativement importants, l'un du 09 juin au 25 août, et l'autre commençant le 1er décembre 1988, prévu jusqu'à début février 1989, ont été programmés.

Dans ce contexte particulier, cinq cycles complets de fonctionnement ont, néanmoins, été réalisés.

## Introduction

This year the Reactor operation programme had to be scheduled in the light of the fuel supply difficulties. Two relatively long shutdowns, one from 9 June to 25 August, and the other starting on 1 December 1988, planned to last until the beginning of February 1989, were scheduled.

It was, however, still possible to complete five operating cycles.

## Einleitung

In diesem Jahr musste das Betriebsprogramm des Reaktors unter Berücksichtigung der Versorgungsprobleme mit Brennelementen aufgestellt werden. Es wurden zwei relativ lange Reaktorstillstände geplant - der eine vom 9. Juni bis zum 25. August, der andere ab 1. Dezember 1988 bis voraussichtlich Anfang Februar 1989.

Trotz dieser besonderen Arbeiten sind fünf volllständige Reaktorzyklen erreicht worden.

# Reactor Operation 1988

### Cycle 1/88

Operation from 5 January to 18 February; the scheduled dates were respected and the cycle was completed without incident.

### Cycle 2/88

Operation from 1 March to 16 April. The cycle was interrupted by two shutdowns. The first was due to a manipulation error, and the shutdown was prolonged by a Xenon poison-out; this was compensated by a two-day extension of the cycle. The second was caused by a mains power failure and was followed by an immediate re-start.

### Cycle 3/88

Operation from 26 April to 9 June. The scheduled dates were respected. Two shutdowns, one caused by the safety circuits of the Cold Source, the other by the release of a safety rod, were both followed by an immediate re-start.

### Cycle 4/88

Operation from 25 August to 8 October. The scheduled dates were respected and the cycle was completed without incident.

### Cycle 5/88

Operation from 18 October to 1 December. The scheduled dates were respected. A single reactor shutdown, occurring during a test of the power measurement circuits, was followed by an immediate re-start.

## Data for 1988

| | |
|---|---|
| Number of days originally scheduled | 220 |
| Actual number of days of operation | 220 |
| Number of equivalent days of full power | 211.64 |
| Actual operating time as proportion of year | 60% |
| Actual operating time in relation to time scheduled | 100% |
| Number of fuel elements used | 5 |
| Number of fuel elements despatched for reprocessing | 4 |
| Number of new fuel elements received | 4 |
| Number of unscheduled shutdowns | 5 |
| including: | |
| brief shutdowns | 4 |
| shutdowns with Xenon poisoning | 1 |

## Operation of the Cold Sources

### Horizontal cold source

The Horizontal Cold Source was installed in beam-tube H5 at the end of 1987.

It started normal operation in the first cycle of 1988, after the authorization of the safety authorities had been obtained.

Its operation was very satisfactory for the whole of the year, apart from a shutdown caused by interference on two pressure sensors in the nitrogen circuit.

### Vertical cold source

No particular comment is called for. It should however be noted that there was a release of low value tritiated deuterium during a test, which had no radiological consequences.

## Helium Refrigerator for the Cold Sources

The new compressor was installed in 1987 and commissioned at the beginning of 1988 with a temporary control system, following problems with the maintenance of the original piston compressors.

During the long summer shutdown, the new control system of the refrigerator was installed, tested and started operation.

The advantages of the new compressor were noted during a brief cut in the mains power supply, which had no effect on the refrigerator, which would not have been the case with the two former compressors.

Furthermore, the new compressor consumes 630 to 650 kW, while the energy consumption was approximately 730 kW with the two piston compressors.

This completes the installation of the equipment financed under the "deuxième souffle".

## Major Events in the Life of the HFR

Advantage has been taken of the reactor shutdowns caused by the delay on new fuel elements to carry out the regular inspection and maintenance specified by our general operation rules.

NUKEM's cessation of operations has aggravated our difficulties as regards the supply of new fuel elements and raises the problem for the future of the search for a new supplier to replace NUKEM and of the building-up of a reasonable reserve stock.

A nuclear safety exercise covering the CEA (Commissariat à l'Energie Atomique) group and the ILL, with the participation of the SCSIN (French nuclear safety authority) was held on the site on 15 June 1988: on the basis of a simulated nuclear accident at the ILL High Flux Reactor, the aim of the exercise was to test the functioning of the arrangements for an emergency.

During the year the general operating regulations (RGE) and the Quality Organisation Manual (MOQ) were updated and forwarded to the Safety Authorities.

## Major Projects Completed or in Preparation

Change of absorber elements of safety rods 1, 2, 3 and 4.

Change of absorber element of the control rod (replacement of BP n° 4 by BP n° 5), its trolley and push rod.

Replacement of the H9 beam-tube thimble.

Replacement of the housing and safety valve of beam-tube H13.

Purchase of 25 tonnes AG3 NET to reconstitute the stock necessary for the production of replacement parts for the reactor (H1-H2, H6-H7, valves, etc).

Acceptance of 4 control rod absorber elements and one push rod.

Start of manufacture of a fuel element support replacement part.

Strengthening of the prestressing in the upper part of the reactor building with additional cables.

Reinstallation in the reactor building of the 'rabbits' (system of irradiation using pneumatic tubes, transferred from the LMA building at CENG).

Acceptance and tests of the high pressure water-jet cutting system (for cutting up radioactive waste).

Commissioning of the controls for the installation of beam-shutters and pumping circuits for new neutron guides from beam-tube H5 (Horizontal Cold Source).

Renewal of the controls of the helium recompression system.

Replacement of all the health physics measuring equipment for the reactor installations.

Replacement and extension of the ILL loudspeaker system.

Replacement of the TCMS 1 computer.

Return to the manufacturer (MTU) and complete overhaul of the emergency Diesel generator n° 1.

Installation of an emergency diesel generator for the HFR Emergency Control Room (PCS).

Modification of the pumping rate adjustment system for the annular space and the associated controls.

Increase in capacity of the industrial water circuit exchangers.

Modifications to conform with fire requirements for the rooms in the centre of building ILL4.

Purchase of a spare main valve for the heavy water circuit.

Purchase of a compressor for the helium recompression system.

Purchase of valves for the effluent gas network to isolate the reactor building.

In 1988 the Detritiation plant treated 11 tonnes of heavy water from FRJ 2 (KFA Jülich), 10 tonnes of heavy water from EL4 (BRENNILIS) and 70 tonnes of heavy water from the RHF.

# Computing Department

# Computing Department

## Introduction

Looking at the list of new systems installed in 1988 one has the impression of solid progress in improving the facilities on offer. The introduction of the VAX 8700, clustered with the 8650 enabled the DEC-10 to be taken out of service, thus creating a homogenous central computing facility. Microvaxes replaced obsolescent or unreliable PDP11s on five instruments. The Ethernet network grew both physically and functionally. And the Macintosh is ubiquitous as a popular and versatile workstation.

Yet despite the evident benefits from these investments, there are some fundamental problems which are clouding the horizon in the medium term. These essentially centre around the role of a Computing Department at a time when almost everyone is either using computers, in some form, or is affected by their presence. Such problems are having to be faced by many organisations, but at ILL the constraints of the staffing policy leave us little room for easily adapting to such a situation. The problem is most apparent as regards support for the Administration Department and in Data Treatment, where the computer users, lacking their own specialists, are requesting more support from the Computing Department than it is able to provide.

In order to go some way to resolve these problems, a number of organisational changes were put into effect during the year, but it is too soon to judge the impact of these. In particular, a major reorganisation of the Department in October came too late to influence the work being reported on here. The structure of this Report is therefore based on the former organisation.

## Introduction

La liste des nouveaux systèmes installés en 1988 donne, en l'examinant, l'impression de gros progrès dans l'amélioration des installations disponibles. L'arrivée du VAX 8700, exploité conjointement avec le 8650, a permis au DEC-10 d'être mis hors-service, créant ainsi une installation centrale homogène d'informatique. Sur cinq instruments, des Microvaxes ont remplacé de vieux PDP11 de fonctionnement incertain. Le réseau Ethernet s'est développé en taille et en fonctions. Le Macintosh également est partout comme un outil à la mode et aux talents polyvalents.

Malgré les avantages évidents retirés de ces investissements, quelques problèmes de fond obscurcissent l'horizon à moyen terme. Ceux-ci tournent essentiellement autour du rôle que joue le Département Informatique à une période où presque tout le monde, soit utilise les ordinateurs d'une manière ou d'une autre, soit s'en trouve influencé. Beaucoup d'organismes doivent affronter de tels problèmes, mais à l'ILL les contraintes de la politique du personnel nous laissent peu de marge de manœuvre pour nous adapter facilement à une telle situation. Le problème est plus apparent en matière de soutien au Département Administratif et en traitement de textes où les utilisateurs d'ordinateurs, ne disposant pas eux-mêmes de spécialistes, sollicitent de la part du Département Informatique plus d'aide qu'il n'est possible d'en fournir.

Dans le but de pallier ces problèmes, un certain nombre de changements dans l'organisation ont eu lieu durant l'année, mais il est trop tôt pour pouvoir juger de l'impact de ceux-ci. En particulier une réorganisation importante du Département en octobre est arrivée trop tard pour avoir une influence sur le travail de l'année 1988. La structure de ce rapport est donc basée sur l'organisation précédente.

## Einleitung

Betrachtet man die Reihe der im Jahr 1988 neu installierten Systeme, so zeigt sich, dass das Angebot an Rechnereinrichtungen erheblich verbessert wurde. Die Einführung einer VAX 8700 gekoppelt mit einer 8650 erlaubte es, die DEC-10 Anlage ausser Betrieb zu nehmen und somit ein homogenes zentrales Rechnersystem zu schaffen. An 5 Instrumenten wurden veraltete oder unzuverlässige PDP11-Rechner durch Microvax Rechner ersetzt. Das Ethernet-Netz wuchs sowohl im Umfang als auch im Funktionsbereich. Macintosh-Rechner sind als beliebte und vielseitige Arbeitsgeräte überall zu finden.

Trotz des offensichtlichen Nutzens dieser Investitionen gibt es grundlegende Probleme, die auf mittelfristige Sicht den Horizont bewölken. Es geht um die tatsächliche Rolle der Abteilung für Computerdienste in einer Zeit, in der fast jeder in irgendeiner Weise Rechner benutzt oder von deren Existenz betroffen ist. Mit dieser Frage haben sich viele Unternehmen zu befassen, am ILL jedoch lassen Einschränkungen in der Personalpolitik kaum Platz für eine einfache Anpassung an solch eine Situation. Am stärksten zeigt sich dieses Problem in der Verwaltung und bei der Datenauswertung, wo Rechnerbenutzer - in Ermangelung ihnen zugeteilter Fachleute - mehr Unterstützung von der Abteilung für Computerdienste verlangen als diese geben kann.

Zur Lösung dieser Probleme wurden im Laufe des Jahres einige Umorganisationen vorgenommen. Es ist jedoch noch zu früh für eine Beurteilung ihrer Auswirkungen. Besonders eine grössere Reorganisation im Oktober fand zu spät statt, um die Arbeiten, über die hier berichtet wird, noch zu beeinflussen. Der folgende Bericht bezieht sich deshalb nur auf die vorherige Organisation.

# Automation and Data Service

## Software A
### (Crystallographic Instruments)

This Group is responsible for software for the instruments D1A, D1B, D2B, D3, D4, D9, D10, D15, D16, D19, D20, DB21, LI5, LI8, S18.

It also has global responsibility for the Ethernet network, and for most DEC-licenced software on VAX-range machines.

The main aim, on the instruments, has been the installation of the three Micro-VAX on D10, D15, and D16. The MAD software was modified to improve standardisation. The results obtained were considered very satisfactory and the performance of the equipment was particularly enhanced. These installations, which took place during the summer reactor shutdown, were successful thanks particularly to a considerable effort by the programmers concerned.

The D4 computer was changed from a PDP11/23 to a PDP 11/73, to improve performance and reliability. The D3 software was completed, and the use of automatic equipment, controlled by the PDP11 via an IEEE link, for the monochromator and the coils, is considered very promising by the personnel concerned. The flipping monitoring system has been completed and can be installed on any ILL instrument as needed.

For the instrument D19, the display software LSTVAX has been completely rewritten on the basis of GKS and has made it possible to define the exact requirements of this instrument for the replacement project planned for 1989.

For the multidetector instruments D9 and D15, the D19 "RETREAT" treatment program has been largely rewritten to adapt it to these instruments and to improve performance.

For the instrument IN10, the SA group has given its support for the provisional version of the VME/OS9 combination.

The ILL local network has been completed: the Ethernet cable is available in all the main ILL buildings, two bridges have been installed, several DECservers are operational, and despite this increase, the availability of the network has been close to 100%. In addition, access to the ILL network has been given to the ESRF computing systems.

## Software B
### (Other Neutron Scattering Instruments)

Software B supports the instruments in the Three-Axis (IN1, INFB, IN3, IN8, IN12, IN14, IN20) and Vercors (IN4, IN5, IN6, IN10, IN11, IN13, D7, D11, D17) Groups.

The major activity for the three-axis instruments was in implementing and testing the standard control program on the new instrument IN14. This has proceeded according to plan, and the first measurements have been successfully completed. New data and program interchange facilities have been incorporated here and on one other spectrometer using Ethernet links to vitiate the absence of fully compatible DECnet on the TSX systems. The CAMAC motor systems on the spectrometers have required further programming effort, especially on IN3.

For the Vercors instruments the first step was made in replacing the PDP11 control computers on the time-of-flight spectrometers by microVAX computers. These greatly simplify the control, on-line surveillance and treatment for the large data volumes engendered by such instruments. The control of IN6 was completely rewritten, re-using a number of standard components from D11 and D17, and was implemented without loss of measuring time. This system will serve as a basis for similar instruments. During the summer shutdown a considerable effort was expended in optimising the control required for the cryostat on IN6, where cryogenic performance has been sacrificed for improved neutron transmission properties. During this period, as a temporary stop-gap a PDP11/24 with extended memory replaced the unreliable PDP11/34 on IN5. A new microVAX was acquired for D7; the first stage of converting data evaluation programs has been completed by the instrument responsible.

The control program of IN13 has been enhanced, and additional continuous monitoring is performed. This instrument was connected to the Ethernet and uses link software identical to the Three-axis spectrometers.

There has been systematic further development of the small-angle scattering spectrometers, with installation of Ethernet hardware, and adoption of IEEE-488 interfaces for additional monitoring and low cost standard control facilities. The control programs have been extended, and data transfer programs and systems updated.

Although not directly within the brief of the Group, the shutdown of the DECsystem10 had a major impact on the general workload. The Vercors instruments with large demands on data-storage and fast printers were constrained to be amongst the last users of the PDP10 for these practical reasons. A major achievement of the Group was to subsequently establish an operating environment on the instrument VAX computers, and on the central cluster which offers transparency to the users. Data access utilities were created by the Group, and directly and indirectly most of the initial data reduction programs were modified in time for the departure of the PDP10.

The Group has contributed to other departmental activities : the use of the Macintosh personal computer has continued to flourish, and a number are now connected in local networks,

and have access to the central VAX cluster as a file-server. A Postscript printer available over the ILL network has animated direct interest in this language; it is used to produce grey-level multidetector image maps very efficiently, as well as line drawings and high grade text output. As in the past the Group has been actively involved in general Graphics facilities, and in internal and external networks.

## Software C
### (Nuclear Physics and Special Instruments)

In the Nuclear Physics area, the Group supports the instruments PN1 (Lohengrin), PN2 (Bill), PN3 (GAMS 1,2,3,4), PN4, PN8 (Cosi fan tutte).

PN1 has acquired a microVAX/GPX and a new data acquisition system based on an Atari PC, which have enabled better on-line data treatment. For the moment the old PDP 11/34 still serves to control the magnetic field but the microVAX should take over completely in the near future.

PN2 has been equipped with its own Benson plotter to speed up the recording of spectra. The PDP 11/23 is now seen as the bottleneck for on-line data inspection.

The set of four g-spectrometers known collectively as PN3 are under constant evolution, which places an ever increasing strain on the computing resources assigned to them. Towards the end of the year funds were made available for a microVAX to relieve this problem.

PN8, another instrument with the need to handle large matrices, is also becoming constrained by the lack of computing power. A solution will have to be found shortly.

The Group's obligations towards the S instruments have always been unclear. At the present time some support is provided for S3, S3B, S20, S20A, S21, S34, TGV, Nessie, .

The most that we can usually do is to encourage the owners of these instruments to follow ILL standards.

The Group also runs a VAX 750, which has traditionally served as a specialised data treatment centre for PN and S instrument users. This machine has recently been integrated into the Central Computer VAX cluster, in order to permit better sharing of peripheral resources.

## Management Information Systems and Office Automation

The Group started the year with a serious problem of priorities to be resolved. Day-to-day support for the two large administration packages, PACHA (personnel) and DEAL (finance) was taking up so much time that it was proving impossible to make any significant progress on other projects.

After examining this matter, the Director decided that the optimum use of the limited staff available would be obtained by their working on those activities which are specific to ILL, such as the visitor programme, with outside assistance being used for the standard commercial packages.

The change-over to this new pattern of working has not been easy. The Administration Department has no computer specialist of its own and finds it inconvenient to rely entirely on outside assistance. But it has enabled serious work to be restarted in other areas. The MISSILL system, which supports the visitor programme and associated activities is to be thoroughly modernized, starting with the Scientific Coordination and Public Relations Office (SCAPRO). A microVAX 3500 has been acquired (to replace a now unreliable PDP 11/34) and the programs will be completely rewritten using up-to-date software tools.

## Computer Maintenance

The evolution of in-house activities in this area is under constant review. The large-scale integration of electronic logic makes it impossible for such components to be repaired locally. Even to run and interpret a diagnostic programme requires considerable understanding of the system.

The general tendency is therefore to have all recently acquired equipment maintained under contract. Even so, there is still important work for ILL technicians, notably in efficiently running the maintenance contracts and in shuffling modules and peripherals between systems, in order to keep down-time to a minimum.

## Telecommunications

Considerable effort has been put into extending the Ethernet local network, which now reaches most parts of ILL, and the ESRF building.

No changes are to be seen in the telephone system, the present exchange having been expanded to the limit of its possibilities. The deficiencies of an analogue-based exchange are beginning to be noticed. It is hoped that a move to a digital exchange can be scheduled before long.

# Central Computing Service

## Replacement of the DEC-10

As already mentioned in the 1987 report, the project to replace the DEC-10 by a VAX 8700 was accepted by the Steering Committee on 26 November 1987.

The closure of the DEC-10 was planned during a reactor shutdown, i.e. for 18 April, and delivery of the VAX 8700 was planned for the end of April. The removal of the DEC-10, which occupied a considerable area, gave the opportunity to rearrange the computer room, which took two weeks. As planned, the VAX 8700 was available on 9 May. It was installed with the VAX 8650 in a homogeneous cluster, i.e. the processors carry out the same type of work, share the same disks (approximately 8 Gigabytes), the same magnetic tape units and the same printers.

As in the previous year the VAX 8650 remains a machine to be shared between ILL and ESRF in the ratio to 2:1.

A VAX 750 was incorporated in this cluster to permit the use of certain specific PDP11 peripherals, and to accept software corresponding to specific applications capable of operating on a single cluster processor (the cost of software licences being lower for this type of computer than for the other two computers in the cluster).

The conversion of the programs was not quite complete when the DEC-10 closed down, but it may be considered that this overlap period for the DEC-10 and VAX 8650 of two years greatly facilitated the changeover. As a first stage the main batch applications were transferred, to take advantage of the power of the new machine. Then when after some months the service had installed the infrastructure of a data base for the experiments, the transfer of the instrument information took place gradually with the availability of treatment for raw data. After the cluster had been set up, the conversion of the programs continued normally and is still continuing, the DEC-10 files being accessible from the VAX machines.

## Operation of the Computing Centre

At the beginning of the year, i.e. for the last three months of its operation, the DEC-10 was still quite popular, with average CPU use of the order of 200 hours per month. During the same period the VAX 8650 reached a maximum saturation, as statistics of use of the order of 600 hours per month were recorded, including more than 350 hours for interactive operation.

The machines operated 24h per day, seven days a week, with the same operator cover as for the previous year, i.e. 16 hours on Monday (5h 30 - 21h 30) and 14 1/2 hours the other 4 days of the week (6h 15 - 20h 45); the rest of the week the machines run in self service operation. Their reliability was such that overall availability was close to 100%.

The Operation Group was very involved in the change of computer:

- reorganisation of the machine room,
- training for management of the cluster,
- development of procedures and tools to improve operation in general, for the organization of safeguards, to facilitate the access to catalogues of back-up files and to certain archive catalogues.

This Group also took responsibility for the operations to safeguard files on word processing and management systems. It was also very concerned with the arrangement of work-rooms for users, and in the renewal and maintenance of terminals and printers provided for all the users (ILL scientists, visitors).

## Systems and Communications

As in 1987, this group continued working on the development of system products essential for recuperation of the DEC-10 archives (programs and data), and to permit the exchange of information with the other centres. On-line documentation has been provided to facilitate use of these products.

The start up of the cluster required major reorganization of the disk space, distribution of batch work and of output spoolers, installation of software and procedures to make the cluster as homogeneous and user friendly as possible, monitoring of the operation of the two systems to permit action to be taken on optimisation parameters and distribution of work. This distribution of work was only possible by incorporating the cluster more closely into the Ethernet network, i.e. by installing some ten DECservers and reducing the number of asynchronous access possibilities.

The handling of instrument data required a particular effort this year to improve and simplify access to information available on line or to archive information. The software was designed to exploit data which will be archived on an optical disk which has been ordered.

The use of the TRANSPAC, JANET, EARN/BITNET networks has considerably increased during this year. The activities of the group, apart from the daily assistance required, had been maintenance of software, installation of new facilities (ex: GMAIL) to facilitate the dispatch of documents to the other international networks, stricter control of access from outside for security reasons, and a more precise identification of the statistics of use to enable each user to appreciate better his expenditure in this area.

The group was also involved in the field of personal computers in collaboration with other members of the Department. As the number of Macintoshes has considerably increased, it was

necessary to organise them in an Appletalk network, to share the printers and to permit access to the cluster. A first network was set up based on machines in self-service operation in the computer building, and now comprises some twenty Macintoshes; it was connected via Ethernet to the cluster in the computing centre to permit easy access to the resources of this cluster, and in particular to share the VAX discs. It will be possible to incorporate the other Appletalk networks in the other buildings into the Ethernet network as necessary.

## Graphics

Here too one of the main tasks of this group has been to make available to the scientists during program conversion phase a number of graphics products written or rewritten to the new standards adopted since the arrival of the VAX 8650: screen plotting to the Tektronix 4014 standard, plots on BENSON, plots on LASER printer, hardcopy from the EVANS & SUTHERLAND workstation on a LASER printer.

As graphic libraries become relatively voluminous and sometimes difficult to manipulate, the Graphics Group has incorporated developments to facilitate use: upgrading by adopting simple precision for graphic calculations, increase in functions and performance (i.e. incorporation of laser printers as output equipment), organisation of sharable libraries, better transparency, particularly for the TEKTRONIX library by recognition of terminal screens.

The recent purchase of a number of workstations, particularly at the instrument level, led us to obtain a new library to treat colour in 2D and 3D applications with drivers interfaced for most graphics equipment. Several of these libraries were tested and one of them, DISSPLA, has just been installed on a GPX station of the Group and the VAX 750 of the cluster, which will thus play the part of library server for the other graphics facilities.

The group thus assists all the scientists in the development of graphics applications, but this role is not limited to cluster applications, but extends to those on the instruments.

## Mathematics

One of the aims of this sector has been to provide assistance to the scientists in the conversion of Fortran programs from the DEC-10 to the VAX. Information notes related to these problems have been circulated. To satisfy the requests of the Crystallography College, the program XTAL has been installed to replace XRAY; adaptations are to be provided to make use of the graphics output on our machines.

In addition, as the workload of the systems group was very heavy this year, the head of this sector assisted with the tests and selection of a product for archiving files. Procedures were developed on the basis of this product for its introduction and to facilitate its use by the users and the operations team.

The **Newsletter** is also distributed by the sector, and another project in progress is the preparation of a "Central Computer User's Guide" for which coordination is necessary between the different members of the Service.

# Administration Department

# Administration Department

## Einleitung

Aus der Arbeit der Verwaltung sind folgende wichtige Ereignisse aus dem Jahre 1988 hervorzuheben:

• Die Zusammenarbeit mit ESRF ist weiter verstärkt worden. Der am 17. Mai 1988 vom ILL und von der ESRF unterzeichnete Pachtvertrag mit dem CEA ermöglicht beiden Instituten die langfristige gemeinsame Nutzung des Forschungsgeländes.

• Beide Institute haben im Interesse der engen wissenschaftlichen und administrativen Zusammenarbeit mit der Ausarbeitung gemeinsamer Dienste und Einrichtungen begonnen. Besonders hervorzuheben ist der inzwischen vom ILL-Lenkungsausschuss und vom ESRF-Rat genehmigte Bau eines Gebäudes für die gemeinsame Bibliothek, für die Theoretiker beider Institute sowie für eine gemeinsame Kantine und eine Cafeteria.

• Die Räumung des künftig der ESRF vorbehaltenen Teils des gemeinsamen Geländes wurde Ende 1988 abgeschlossen, so dass die Bauarbeiten zur Errichtung der ESRF-Anlage Anfang 1989 fristgerecht beginnen können.

• Wie auch in den vergangenen Jahren hat das ILL der ESRF gegen Erstattung der damit verbundenen Personalkosten Verwaltungshilfe geleistet, insbesondere in den Bereichen Personalwesen, Reisekosten, Einkauf, Buchhaltung, Sprachkurse, Schulwesen.

• Am 13. Mai 1988 wurde zwischen dem ILL und der Schweiz eine Vereinbarung über die wissenschaftliche Beteiligung am ILL unterzeichnet. Die Vereinbarung ermöglicht der Schweiz die wissenschaftliche Nutzung der ILL-Einrichtungen nach denselben Grundsätzen, wie sie für Wissenschaftler aus den drei Mitgliedsstaaten gelten. Die Schweiz leistet dafür einen Zuschuss zum ILL-Haushalt in Höhe von 1,5 % der Gesamtausgaben. 1987 war bereits eine entsprechende Vereinbarung mit Spanien in Kraft getreten.

• Die Anfang des Jahres angespannte Situation bei der Versorgung mit hochangereichertem Uran und Brennelementen hat sich Ende 1988 - nach dem Eintreffen der aus den USA bestellten 77 kg im Oktober 1988 - wesentlich verbessert. Weitere 76 kg hochangereichertes Uran aus den USA werden im Frühjahr 1989 erwartet. Nach dem Ausscheiden von NUKEM als Hersteller von Brennelementen werden die zukünftigen Elemente von CERCA geliefert werden. Die aufgetretenen Versorgungsschwierigkeiten führten dazu, dass 1988 lediglich 5 Reaktorzyklen durchgeführt wurden. 1989 sind 6 Zyklen vorgesehen. Zudem soll in den Jahren 1989 bis 1991 eine Reserve von insgesamt 3 Brennelementen angelegt werden, um eventuellen Versorgungsschwankungen besser begegnen zu können.

• Der Lenkungsausschuss tagte im Juni 1988 in Grenoble und im November 1988 auf Einladung des deutschen Gesellschafters im Reichstagsgebäude in Berlin. Dort wurde das ILL-Budget 1989 mit Gesamtausgaben in Höhe von 301,80 Mio F (Geldwert 1989; einschl. Übertrag) beschlossen.

• Im Juni 1988 verliess der bisherige Leiter der Verwaltungs- und Finanzabteilung, Christoph Eitner, das ILL und übernahm eine neue Funktion im Bundesministerium für Forschung und Technologie in Bonn. Sein Nachfolger wurde Hans-Martin Spilker.

## Introduction

Dans le domaine de l'Administration, les faits suivants méritent d'être mentionnés pour l'année 1988 :

• La collaboration avec l'ESRF s'est poursuivie et a été renforcée. Le contrat de bail signé le 17 mai 1988 entre l'ILL et l'ESRF avec le CEA permettra aux deux instituts d'utiliser à long terme et en commun le site commun de recherche.

• Les deux instituts ont commencé à mettre sur pied des services et dispositifs communs, ceci dans l'intêrêt d'une étroite collaboration scientifique et administrative. Il faut souligner que la construction d'un bâtiment qui abritera la bibliothèque commune, les théoriciens des deux instituts ainsi que la cantine commune et la cafétéria, a reçu entre-temps l'autorisation du Comité de Direction de l'ILL et du Conseil de l'ESRF.

• La libération de la partie du site dont l'usage sera, à l'avenir, réservé à l'ESRF, a été terminée fin 1988 de sorte que les travaux de construction des installations de l'ESRF pourront commencer, comme prévu, au début de l'année 1989.

• Comme les années précédentes, l'ILL a apporté une aide administrative, plus particulièrement dans les domaines personnel, frais de mission, achats, comptabilité, cours de langue, Ecole, contre remboursement des frais de personnel correspondants.

• Le 13 mai 1988, une convention a été signée entre l'ILL et la Suisse sur la participation scientifique de ce pays à l'ILL. Cette convention permettra à la Suisse d'utiliser les dispositifs scientifiques de l'ILL selon les mêmes principes que ceux appliqués aux chercheurs des trois pays membres. En contre-partie, la Suisse versera une contribution au budget de l'ILL équivalente à 1,5 % du volume de dépenses totales. Une convention analogue avait déjà été signée avec l'Espagne en 1987.

• En ce qui concerne l'approvisionnement en uranium très enrichi et en éléments combustibles, la situation tendue en début d'année s'est beaucoup améliorée avec la livraison au mois d'octobre 1988 des 77 kg d'uranium enrichi en provenance des Etats-Unis. Une autre livraison de 76 kg d'uranium très enrichi également en provenance des Etats-Unis est attendue au printemps 1989. A la suite de la cessation d'activité de la société NUKEM, un des fournisseurs de l'ILL en éléments combustibles, ceux-ci seront à l'avenir fournis par CERCA. Ces difficultés d'approvisionnement ont eu pour conséquence qu'en 1988, le Réacteur n'a fonctionné que pendant 5 cycles. 6 cycles sont prévus en 1989. L'ILL reconstituera au cours des années 1989 à 1991 une réserve de 3 éléments combustibles au total ; il sera ainsi en mesure de mieux faire face à d'éventuelles fluctuations d'approvisionnement.

• Le Comité de Direction s'est réuni au mois de juin 1988 à Grenoble et, sur invitation de l'Associé allemand, au mois de novembre 1988 à Berlin dans les bâtiments du Reichstag. C'est là que fut adopté le budget de l'ILL avec un volume total de dépenses de 301,80 Millions F (valeur monétaire 1989, transferts inclus).

• Au mois de juin 1988, le Chef du Département Administratif et Financier, M. Christoph Eitner, a quitté l'ILL pour prendre de nouvelles fonctions au Ministère Fédéral de la Recherche et de la Technologie à Bonn. Il a eu pour successeur M. Hans-Martin Spilker.

# Introduction

The following important items in the work of the Administration in 1988 may be noted:

• Collaboration with the ESRF has been further enhanced. The contract of lease signed on 17 May 1988 by the ILL and ESRF with the CEA provides for the long-term joint use of the research site by the two institutes.

• In the interest of close scientific and administrative cooperation the two institutes have started work on the preparation of joint services and facilities. Particular reference should be made to the construction of a building for the joint library, the theoreticians of the two institutes and for a joint canteen and cafeteria, which has now been approved by the ILL Steering Committee and the ESRF Council.

• The clearance of the part of the common site reserved in future for the ESRF was completed at the end of 1988, so that the construction work on the ESRF facilities can start on schedule at the beginning of 1989.

• As in the previous year ILL has provided ESRF with administrative assistance against reimbursement of the associated staff costs, in particular in the fields of personnel, travel, purchasing, accounting, language courses and schooling.

• On 13 May 1988 an agreement was signed by ILL and Switzerland covering Swiss scientific participation at ILL. The agreement provides for Swiss scientific use of the ILL facilities on the same principles as for scientists from the three member countries. In return Switzerland pays a contribution to the ILL budget of 1.5% of total expenditure. A similar agreement with Spain took effect in 1987.

• The difficult situation at the beginning of the year as regards the supply of highly enriched uranium and fuel elements had considerably improved by the end of 1988, after the arrival in October 1988 of 77 kg uranium ordered from the USA. A further 76 kg are expected from the USA in spring 1989. Now that NUKEM is no longer available as a manufacturer of fuel elements, the future elements will be supplied by CERCA. The supply difficulties resulted in the number of cycles in 1988 being reduced to 5. For 1989 six cycles are planned. In addition a reserve of 3 fuel elements is to be built up during the years 1989 to 1991, in order to cope better with any fluctuations in supplies.

• The Steering Committee met in June 1988 in Grenoble, and in November 1988 at the invitation of the German Associate in the Reichstag building in Berlin. There the ILL Budget for 1989 was adopted with total expenditure of 301.8 MF (1989 prices; incl. carry forward).

• In June 1988 the previous Head of the Administration and Finance Department, Christoph Eitner, left ILL to take up a new post at the Federal German Ministry for Research and Technology in Bonn. His successor is Hans-Martin Spilker.

# Finance

## Implementation of 1988 Budget

The budget authorized for 1988, including 13 958 KF not utilized in 1987 and brought forward, amounted to a total of 298 558 KF excluding taxes. This was financed as follows:

| Budget breakdown | KF |
|---|---|
| Balance of expenditure 1987 | 3,600 |
| ILL's own income | 8,470 |
| Spanish and Swiss contributions | 8,540 |
| Brought forward from 1987 | 13,958 |
| Associates' contributions | 263,990 |
| **Total budget** | **298,558** |

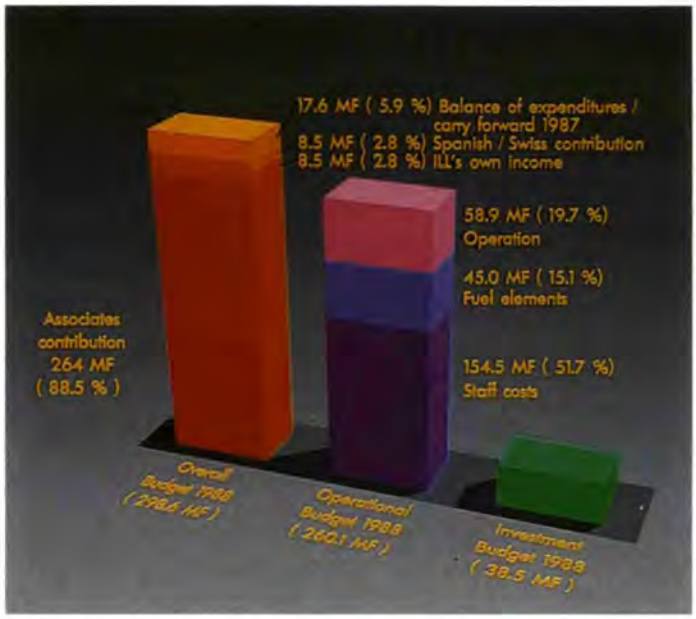



*Figure 114 The 1988 Budget of the ILL*

In addition the French Associates made available a special credit of 4.5 MF for the clearance of the ESRF site. This special credit was used in particular for the construction of a new building for the Works Committee, the transfer of several buildings and the modification of the drainage system.

Because of delays in uranium deliveries and, consequently, in the production of fuel elements, it was only possible to run five reactor cycles in 1988.

Regarding the scientific programme, 1988 was characterized by the commissioning of the Horizontal Cold Source, the

### Comparison of 1987 and 1988 Normal Budgets (Expenditure)

| Expenditure | Expenditure 1987 | | 1988 Budget excl. carry forward from 1987 | | Carry forward from 1987 | Revised 1988 Budget | |
|---|---|---|---|---|---|---|---|
| | MF | % | MF | % | MF | MF | % |
| 1) Staff costs | 148.10 | 55.0 | 154.50 | 54.3 | | 154.50 | 51.7 |
| Other exp. on personnel | 2.10 | 0.8 | 1.70 | 0.6 | | 1.70 | 0.6 |
| 2) Fuel Elements | 23.20 | 8.6 | 35.40 | 12.4 | 9.60 | 45.00 | 15.1 |
| Consumables | 27.20 | 10.1 | 29.60 | 10.4 | | 29.60 | 9.9 |
| Long term supplies and services | 7.90 | 2.9 | 8.30 | 2.9 | | 8.30 | 2.8 |
| Short term supplies and services | 11.30 | 4.2 | 10.80 | 3.8 | | 10.80 | 3.6 |
| Transport | 2.10 | 0.8 | 1.80 | 0.6 | | 1.80 | 0.6 |
| Misc. costs | 6.40 | 2.4 | 7.00 | 2.5 | | 7.00 | 2.3 |
| Taxes and Fees | 1.50 | 0.5 | 1.40 | 0.5 | | 1.40 | 0.5 |
| 3) Operation | 56.40 | 20.9 | 58.90 | 20.7 | | 58.90 | 19.7 |
| I. Total operation | 229.80 | 85.3 | 250.50 | 88.0 | 9.60 | 260.10 | 87.1 |
| Buildings | 2.50 | 1.0 | 0.60 | 0.2 | | 0.60 | 0.2 |
| Equipment | 6.60 | 2.4 | 4.50 | 1.6 | 2.30 | 6.80 | 2.3 |
| Instruments | 19.60 | 7.3 | 18.20 | 6.4 | 1.40 | 19.60 | 6.6 |
| Other investments | 10.90 | 4.0 | 10.80 | 3.8 | 0.70 | 11.50 | 3.8 |
| II. Investments | 39.60 | 14.7 | 34.10 | 12.0 | 4.40 | 38.50 | 12.9 |
| **III. Total expenditure** | **269.40** | **100.0** | **284.60** | **100.0** | **14.00** | **298.60** | **100.0** |
| IV Clearance site ESRF | | | | | | 4.50 | |

## Comparison of 1987 and 1988 Normal Budgets (Income)

| Income | Income 1987 | | Budget 1988 excl. carry forward | | Revised Budget 1988 | |
|---|---|---|---|---|---|---|
| | MF | % | MF | % | MF | % |
| Balance of expenditure | | | 3.60 | 1.3 | 3.60 | 1.20 |
| ILL's own income | 10.90 | 4.1 | 8.50 | 3.0 | 8.50 | 2.80 |
| Spanish and Swiss contributions | 4.10 | 1.5 | 8.50 | 3.0 | 8.50 | 2.80 |
| Associates' contributions | 254.40 | 94.4 | 264.00 | 92.7 | 264.00 | 88.50 |
| Carry forward 1987 | | | | | 14.00 | 4.70 |
| **Total** | **269.40** | **100.0** | **284.60** | **100.0** | **298.60** | **100.00** |
| Clearance site ESRF | | | | | 4.50 | |

Neutron-Antineutron Experiment and the spectrometer IN14, and by the continued development and construction of the instruments IN15, IN10C and D22 at the Horizontal Cold Source.

# Forward Look

The ILL's medium-term investment plans in the scientific field are essentially as follows:

- the completion of the instruments on the Horizontal Cold Source (IN10C, D22, IN15) with an investment of 6.2 MF in 1989 and 2.6 MF in 1990;
- the improvement of instruments and the replacement of old instruments (3.2 MF in 1989, 5.4 MF in 1990 and 8 MF p.a. from 1991 to 1993);
- the replacement of equipment necessary for the safety and reliable performance of the reactor: beam tubes H1-H2, H6-H7, safety rods,etc.;
- the construction of a common building with ESRF;
- the build-up of a reserve of three fuel elements within the next three years.

Furthermore the Institut has started preparing a concept for a new Modernisation Programme for the instruments and the reactor for the years after 1992.

# Purchasing

Severe problems were encountered in procuring uranium; the delivery of 77 kg due in 1986 finally arrived this year. Two orders for 35 kg and 76 kg of highly enriched uranium were placed with COGEMA, in 1988. For future fuel element production, raw outer and inner cylinders were ordered from VAW. New equipment for cutting irradiated parts using a high pressure water jet was purchased from Weller Engineering Ltd.

Major orders were placed with FORGEAL, CEGEDUR and VAW for several tonnes of aluminium alloy AG 3 NET which is the raw material for future fabrication of replacement parts for the reactor, including coupling sleeves, beam tube liners for H1, H2, H6 and H7.

Further progress was made in the construction of the new instruments of the Horizontal Cold Source; IN14 is nearing completion; IN10C will be finished in 1989; the collimation system for the small angle spectrometer D22 was ordered from SIGRI.

Other major purchases for the instruments include two cryostats with cooling systems for D10 which will be made at the CENG and the detector for IN5 to be manufactured by PECHINEY XERAM.

Several instrument computers were replaced by taking advantage of a special offer from DIGITAL who supplied Microvax II computers with 40% discount on catalogue price and trade-in of the old PDP11 computer. Three Microvax's were thus acquired for D10, D15 and D16. Vax stations were purchased for IN15 and D7. A Vax 3500 has been ordered for future Management Information Systems regarding visiting scientists, library and travel. New maintenance contracts were also negotiated with DIGITAL and a 35% discount on cost was obtained in return for a reduced response time on some of the less critical systems.

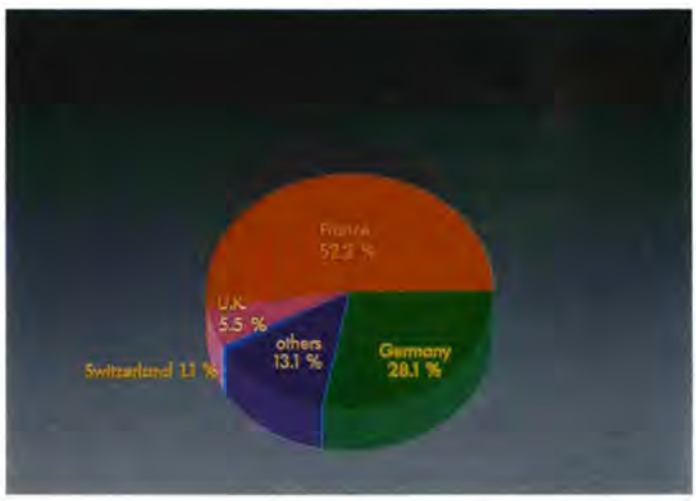



*Figure 115 The breakdown of 1988 purchases in the various countries.*

# Administration Department

The old works committee building has been demolished in order to clear the site for construction of the ESRF. Consequently, a new building to house the works committee was built by ROYANS TRAVAUX.

The ILL continues to handle orders for the ESRF where the value of contracts placed (committments) is approaching the level of the ILL orders in 1988.

Considerable savings were achieved for both organisations by competitive tendering for major purchases, as well as by negotiation with regular suppliers for routine orders.

The distribution of ILL's free purchases* in 1988 is shown in the diagram (10 months only).

| | MF | % |
|---|---|---|
| France | 14.3 | 52.2 |
| UK | 1.5 | 5.5 |
| Germany | 7.7 | 28.1 |
| Switzerland | 0.3 | 1.1 |
| Others | 3.6 | 13.1 |
| **Total** | **27.4** | **100** |

* Free purchases means that a free choice of suppliers was possible excluding therefore the fuel cycle, electricity and small purchases of less than FF 50,000.

## Stores

All deliveries of purchased equipment were received and checked quantitatively by the storemen who at the same time carried out the despatch of samples, returned equipment, etc.

A number of new items were added to the range of articles held in stock and some of the less popular articles were removed reflecting the changing needs of scientists, technicians and other staff.

# Personnel

## Staff

The reduction of the staff complement by 17.5 posts after completion of the Modernisation Programme was achieved at the end of 1987. In 1988 the authorized staff complement remained at 493 posts, including two Spanish thesis students. Of these 493 posts, 490 were occupied on 31 December 1988.

| | | |
|---|---|---|
| French | 66.5% | (65.8%) |
| German | 13.7% | (14.4%) |
| British | 13.3% | (13.8%) |
| Others | 6.5% | ( 4.0%) |

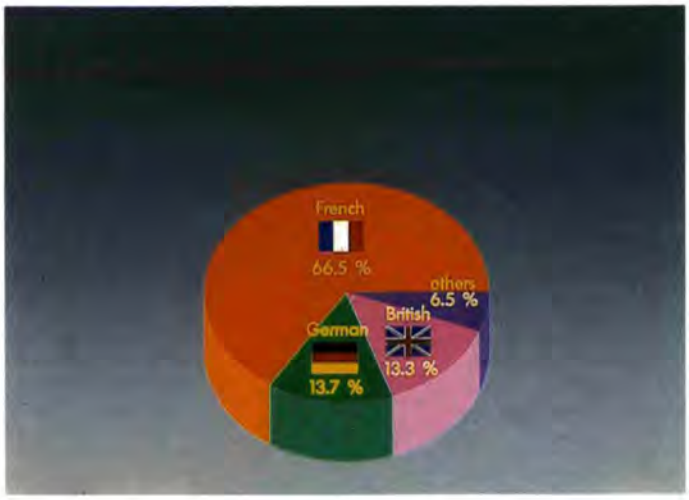



*Figure 116 Staff breakdown by nationality in 1988*

The table above illustrates the percentage of staff of different nationalities at the ILL (in brackets the corresponding figures for 1987).

There was a slight increase in the number of staff of French and 'other' nationalities in 1988 in comparison with 1987. The percentage of German and British staff declined by a corresponding amount. As in 1987 the Institut had difficulties, particularly as regards technicians and other "non-cadres", in recruiting German and British staff who meet its requirements and can be incorporated in the ILL salary scales. The Steering Committee and the Management agree that measures should be taken to achieve a better balance between staff from the three Member States.

### Staff Movements and Posts in 1988

| Categories | Position on 31.12.87 | | | | | Departures 88 | | | | | Recruitment 88 | | | | | Difference | | | | | Position on 31.12.88 | | | | |
|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|---|
| | G | B | F | O | T | G | B | F | O | T | G | B | F | O | T | G | B | F | O | T | G | B | F | O | T |
| **Cadres** | | | | | | | | | | | | | | | | | | | | | | | | | |
| Scientists (perm) | 12 | 7 | 7 | 3 | 29 | - | 1 | 1 | - | 2 | - | - | - | 2 | 2 | - | -1 | -1 | 2 | - | 12 | 6 | 6 | 5 | 29 |
| Engineers | - | - | 2 | - | 2 | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | - | -2 | - | 2 |
| Scientists (non-perm) | 11 | 8 | 12 | 9 | 40 | 1 | 4 | - | 4 | 9 | 3 | 4 | 3 | 4 | 14 | 2 | - | 3 | - | 5 | 13 | 8 | 15 | 9 | 45 |
| **Total** | 23 | 15 | 21 | 12 | 71 | 1 | 5 | 1 | 4 | 11 | 3 | 4 | 3 | 6 | 16 | 2 | -1 | 2 | 2 | 5 | 25 | 14 | 23 | 14 | 76 |
| **Other "cadres"** | 13 | 8 | 43 | - | 64 | 1 | 1 | 2 | - | 4 | 1 | 1 | 1 | - | 3 | - | - | -1 | - | -1 | -13 | 8 | 42 | - | 63 |
| **Total "cadres"** | **36** | **23** | **64** | **12** | **135** | **2** | **6** | **3** | **4** | **15** | **4** | **5** | **4** | **6** | **19** | **2** | **-1** | **1** | **2** | **4** | **38** | **22** | **65** | **14** | **139** |
| **Thesis students** | 9 | 8 | 6 | 3 | 26 | 4 | 5 | 3 | - | 12 | 3 | 4 | 6 | 2 | 15 | -1 | -1 | 3 | 2 | 3 | 8 | 7 | 9 | 5 | 29 |
| **"Non-cadres"** | | | | | | | | | | | | | | | | | | | | | | | | | |
| Technicians | 16 | 26 | 123 | 2 | 167 | 3 | 1 | 4 | - | 8 | - | - | 3 | - | 3 | -3 | -1 | -1 | - | -5 | 13 | 25 | 122 | 2 | 162 |
| Others | 9 | 10 | 127 | 12 | 158 | 1 | - | 2 | 1 | 4 | - | 1 | 5 | - | 6 | -1 | 1 | 3 | -1 | 2 | 8 | 11 | 130 | 11 | 160 |
| **Total "Non-cadres"** | **25** | **36** | **250** | **14** | **325** | **4** | **1** | **6** | **1** | **12** | **-** | **1** | **8** | **-** | **9** | **-4** | **-** | **2** | **-1** | **-3** | **21** | **36** | **252** | **13** | **322** |
| **Grand total** | **70** | **67** | **320** | **29** | **486** | **10** | **12** | **12** | **5** | **39** | **7** | **10** | **18** | **8** | **43** | **-3** | **-2** | **6** | **3** | **4** | **67** | **65** | **326** | **32** | **490** |

G = German, B = British, F = French, O= Other, T = Total

# Administration Department

*Mr. A. Vasquez, better known by his nickname of Chiquilin, retired after 18 years of work at ILL, almost 700,000 km without an accident at the wheel of cars always beautifully maintained. The occasion was marked by a small celebration with some of his friends and colleagues. Mr. H. M. Spilker, Head of the Administration, summarized his long career. The photograph shows Mr. Vasquez opening the present from his colleagues.*

The average age increased from 40.79 in 1987 to 41.73 in 1988 (42.65 for men, 40.80 for women). The breakdown by sex remained essentially the same (83.5% men, 16.5% women).

## Collective Agreement

Notice was given by the Director on 27 April 1988 to terminate the ILL Collective Agreement, after previous negotiations with the unions on amendments to the present agreement, in particular as regards the Productivity Bonus, had proved unsuccessful. According to the terms of the Collective Agreement it will remain in force until 29 June 1989. Negotiations with the Unions are under way to agree on a new Collective Agreement.

## Salaries

There was an overall salary increase of 2.2% in 1988. Advancements represent 1.5% and seniority 0.35% of the total payroll.

## Guest Scientists

Approximately 1860 visits were made by guest scientists to the ILL between January and November 1988. 30.7% of the visitors were from laboratories in France, 25.8% from Germany, 24.3% from the U.K. and 19.2% from various other countries.

## ILL/ESRF Collaboration

The ILL's Personnel Section continues to help ESRF as regards travel expenses for ESRF staff and visitors as well as management of pay and holidays, while ESRF is establishing its own units for these functions. In December 1988 this service covered approximately 60 employees on the ESRF payroll.

# Welfare and General Services

## Housing

### Employer's contribution to housing

The easing of the housing situation in Grenoble, referred to last year, has continued. Rents on the free market have however remained high. Staff accordingly seek housing at reasonable cost (HLM type): ten families obtained accommodation in 1988, with the aid of legally required contributions from ILL to housing organizations (average figure: 50,000 F).

Since 1980 ILL has financed forty flats or houses in this way.

Thirteen loans have also been obtained for staff members through organizations collecting contributions from employers. The average loan was 60,000 F, at a very low interest rate (1%).

Since 1971 270 staff members have received such loans.

### New families

Thirty ILL or ESRF staff or long-term visitors have been helped to find accommodation in the private sector.

The 20 ILL furnished flats have been let to 112 ILL or ESRF scientists for short periods.

## International Education

This year the ILL has continued its support for international education in Grenoble: all the teachers in the German and English "sections" in the Houille Blanche Primary School and the Eaux-Claires and Stendhal Secondary Schools are employed on ILL contracts until 31 August 1989, and are accordingly paid by the ILL.

The German government reimburses the expenditure on the German teachers. For the British teachers, the ILL receives contributions from ESRF, the Council of the Isère Department and the City of Grenoble.

Discussions are in progress with a view to incorporating the international sections completely in the French national education system with effect from September 1989.

The Houille Blanche Primary School has approximately 160 pupils, including 20 German and 41 English-speaking children.

The Lycée International Stendhal has four "international sections": German, English, Spanish, Italian in the first two classes (children aged 11 to 13) and in "seconde" and "première" (age 15 to 17). At the end of 1988 there were 27 German and 57 English-speaking children at the Lycée International Stendhal. By autumn 1990 there will be international classes at all levels. Teachers are already preparing their pupils for the examinations of the country of origin ('0' and 'A' Levels, Abitur, etc.). Numerous contacts have been made with international schools, particularly in Germany and Italy.

## Training

Requests for training are generally following technological developments. Thus in 1988 there has been an increase in requests for training on Macintosh software, C.A.D., advanced communication techniques, etc.

In general the Grenoble training organizations can satisfy the requirements. In technical fields, some ILL scientists and engineers assist the Training Office in the preparation of specific courses designed to cover problems encountered.

Two young employees are spending two academic years training as engineers on courses paid for by "FONGECIF".

Six French courses at various levels have been organized by the Training Office. These are attended by 75 to 80 non-French staff or guest scientists at ILL, ESRF, Max Planck, CNRS, etc.

# Communications

# College 2

## Theory

"Strong coupling theory of high $T_c$ superconductors."
D. RAINER, University of Bayreuth.

"Superconductivity in a narrow band system with intersite electron pairing."
R. MICNAS, CRTBT, Grenoble.

"Soft and hard turbulence in a Rayleigh-Bénard experiment."
S. THOMAE, IFF, KFA Jülich.

"L'échange quantique dans $^3$He."
H. GODFRIN, ILL.

"Propriétés élastiques des matériaux hétérogènes: les gels de silice."
R. MAYNARD, CRTBT, Grenoble.

"Quantum dynamics of weakly coupled superconductors."
U. ECKERN, University of Karlsruhe.

"Diffusion of small particles in glasses."
W. SCHIRMACHER, Technical University of München.

"Relevance of classical chaos in quantum mechanics."
G. CASSATI, Université de Milan.

"La haute température critique de $YBa_2Cu_3O_7$ est-elle explicable par une théorie métallique ?"
R. COMBESCOT, Ecole Normale Supérieure, Paris.

"Fluctuations quantiques dans une généralisation de la théorie du champ moyen. Application à la théorie du BCS."
P. SCHUCK, ISN, Grenoble.

"Lattice gauge theories: an overview for non-specialists."
J. JERSAK, Aachen.

"Lattice pinning in change density wave systems."
S. AUBRY, Laboratoire Léon Brillouin.

"The strong coupling polaron mobility."
A. GOGOLIN, Institut Landau, Moscou.

"Experiments on spin polarized $^3$He-$^4$He mixtures."
R. BOWLEY, University of Nottingham and ILL.

"Correlations in the Gutzwiller wavefunction: Exact results in one dimension."
F. GEBHARD, T. H. Aachen.

"Broken particle-hole symmetry in critical fluids."
R. GOLDSTEIN, Physique Théorique, Saclay.

"Les propriétés statiques et dynamiques du modèle Heisenberg à deux dimensions."
D. GREMPEL, ILL.

"Quelques aspects de la théorie des supraconducteurs à haute $T_c$."
M. LAVAGNA, Laboratoire Louis Néel et ILL.

"Structure électronique, corrélations et supraconductivité dans les nouveaux matériaux à haute température critique."
D. MAYOU, LEPES, CNRS.

"First principle approach to tight binding methods."
F. FLORES, Université de Madrid.

"Comment comprendre la mécanique quantique."
R. OMNES, Université Paris-Sud.

"Structural aspects and stability of quasicrystals."
M. JARIC, Texas A/M University.

"First principle approach to tight binding methods."
F. FLORES, Madrid.

"Topological excitations in 2-d antiferromagnets."
J. RODRIGUEZ, ILL.

"Quelques remarques sur l'hydrodynamique des suspensions."
P. NOZIERES, ILL.

"Wave propagation in random media: fluctuations in transmission."
B. SHAPIRO, Technion (Israel).

"Squeezed polarons in 1D CDW states."
Dr. ZHENG HANG, Université Jiao Tong, Shangaï (LEPES).

"Anderson localisation and the quantum hall effect."
J. T. CHALKER, University of Southampton.

"Weak coupling theory of the Hubbard model and high $T_c$ superconductors."
I. D. DZYALOSHINSKI, Institut Landau, Moscou.

"Neutral fermions in antiferromagnets."
I. D. DZYALOSHINSKI, Institut Landau, Moscou.

"Soft mode cristallization."
I. D. DZYALOSHINSKI, Institut Landau, Moscou.

"Quantification stochastique et nouveau principe variationnel en théorie des champs."
P. GRANGE, ILL.

"Electronic properties of high $T_c$ superconductors."
C. A. BALSEIRO, Bariloche et ILL.

"Defect core structure in nematic crystals."
N. SCHOPOHL, ILL.

"The impurity pinning in quasi-one dimensional superconductors."
Y. SUZUMURA, Tohoku University et Orsay.

"New theory of strong coupling superconductivity and high $T_c$ superconductivity."
A. S. ALEXANDROW, Moscow Physical Engineering Institute.

"Vacancies in 2nd quantum antiferromagnets."
E. SIGGIA, CORNELL et ENS, Paris.

"Computer studies of 2nd liquid crystals."
P. HOLDSWORTH, ILL.

"Are metal-oxide superconductors bosonic (bipolaronic) superfluids?"
L. J. DE JONGH, kam. Onnes Inst., Leiden.

"Mesoscopic fluctuations in small disordered conductors."
B. ALTSHULER, Leningrad.

"Cooper instability in spin liquids."
N. ANDREI, Rutgers University.

"Stability de l'antiferromagnétisme pour les systèmes de spins quantiques à 2 dimensions."
B. DOUCOT, CRTBT.

"Antiferromagnétisme et supraconductivité dans les conducteurs organiques."
C. BOURBONNAIS, Univ. Sherbrooke et Orsay.

"Conflit entre instabilité temporelle et instabilité spatiale : Dynamique non linéaire des matériaux irradiés."
J. VILLAIN, CEN-Grenoble

# College 3

## Fundamental and Nuclear Physics

"New developments in Ge detector technology."
A. SCHÖDER, Univ. Köln.

"Proton neutron interactions and collectivity in odd-odd nuclei."
J. VAN MALDEGHEN, INW Gent.

"The Livermore neutrino experiment."
W. STÖFFL, Lawrence Livermore National Laboratory.

"Corrections to the nuclear mean-field: a non relativistic problem only?"
P. GRANGE, ILL.

"First application of Fermion dynamical symmetry to nuclear structure."
J. DUKELSKY, Strasbourg.

"The characteristics of the fission fragments from the fission of selected Pu isotopes."
P. SCHILLEBEECKX, CBNM.

"Meson exchange currents and electromagnetic properties of nuclei."
J. F. MATHIOT, CERN.

"Berry's phase and applications in rotating nuclei."
P. RING, München.

"The role of symmetry in odd-A nuclei."
D. WARNER, NSF, Daresbury.

"Determination of the neutron lifetime by trapping of decay protons."
J. BYRNE, Sussex.

Review seminar: "Fundamental physics with ultracold neutrons."
W. DREXEL, ILL.

"Astrophysical implications of recent experiments on $^{176}$Lu."
N. KLAY, Karlsruhe.

"A microscopic approach to low energy nuclear spectroscopy."
P. QUENTIN, Bordeaux.

"Making light work of ultratrace analysis."
K. LEDINGHAM, Glasgow.

"Nuclear physics constraints in bringing the astrophysicals r-process to the waiting point."
A. WOEHR, Mainz.

"Microdosimetry in radiation protection."
B. MORE, National Physics Laboratory.

"Search for resonances in $e^+e^-$ scattering."
K. SCHRECKENBACH, ILL.

"Automatic analysis of gamma-ray spectra."
G. G. COLVIN, AERE Harwell.

"Parity and time-inversion violation in nuclear reactions."
V. BUNAKOV, MPI, Heidelberg.

"Spectroscopy of laser-cooled ions: high resolution spectroscopy Coulomb clusters."
W. ITANO, NBS.

"The $^3$He(n,$\gamma$) cross-section and the solar neutrino problem."
F. WOLFS, Argonne National Laboratory.

"Coulomb crystals in storage rings."
R. HASSE, GSI.

"Low temperature quantum properties of spin-polarized $^3$He."
M. LEDUC, Laboratoire de Spectroscopie Hertzienne de l'ENS.

Review seminar: "GAMS 1,2,3,4. . . ."
H. BORNER, J. JOLIE, S. ROBINSON (ILL), M. S. DEWEY (NBS).

"Short lifetimes measured by DSA method for tests of shell-model states in the sd-shell nuclei."
J. KEINONEN, University of Helsinki.

"Shape coexistence and intruder states in even-even nuclei."
K. HEYDE, Ghent.

# College 4

## Structural and Magnetic Excitations

"Anharmonic effects in rare gas solids: experiment and theory."
J. ECKERT, Los Alamos and ILL.

"Structures and phase transitions of Xe and $D_2$ on MgO."
D. DEGENHARDT, University of Kiel and ILL.

"Theoretical possibilities of type 1 magnetism in FCC-systems."
M. LONG, Rutherford Laboratory.

"Polarized neutron studies of magnetic excitations in the one-dimensional antiferromagnet TMMC."
R. PYNN, Los Alamos.

"Dynamical properties of metallic reentrant spin-glasses studied by inelastic neutron scattering."
S. LEQUIEN, Léon-Brillouin, Saclay.

"Collective excitations in liquid cesium."
T. BODENSTEINER, Garching, München.

"Hall conductance and band structure of a 2D electron gas in a periodic potential and strong magnetic field."
F. AXEL, Laboratoire de Physique des Solides, Orsay and Lyman Laboratory, Havard University.

"Experiments with fast neutrons."
W. WASCHKOWSKI, Garching, München.

"Incommensurate phases studied by a computer simulation."
K. PARLINSKY, Institute of Nuclear Physics, Krakow (Poland).

"Why has $PrBa_2Cu_3O_7$ no superconductivity ?"
E. HOLLAND-MORITZ, University of Köln.

"Multilayers of TiNi and SiGe: X-ray reflection measurements near and at total reflection."
T. MÖLLER, University of Kiel.

"Magnetic excitations in the quasi one-dimensional antiferromagnetic singlet groundstate system $CsFeBr_3$."
B. DORNER, ILL.

"Magnetic structure and dynamics of reentrant spin glasses."
R. ERWIN, NBS Washington.

"Disorder phenomena in quasi 2D-magnetic systems: neutron scattering, magnetization measurements and magnetic resonance in $Rb_2MnxCr_{1-x}Cl_4$."
R. GEIK, Universität Würzburg.

"Hydrogen in metals: Synthesis of new hybrides of transition metals at high pressure of $H_2$, Phase diagram, Structures, Magnetic properties, Superconducting properties."
E. G. PONYATOVSKII, High Pressure Physics Laboratory, Chernogolovka (USSR).

"Frozen phonon calculations for transition metals."
K. M. HO, Inst. für Nukleare Festkörperphysik, Karlsruhe.

"A synthetic scattering law for slow neutron - molecule interaction."
J. R. GRANADA, Centro Atomico Bariloche (Argentine).

"NMR investigation of the proton glass behaviour in RbDP-ADP mixed crystals."
V. H. SCHMIDT, Montana State University.

"Helium scattering studies of the dynamics of a Xe (Kr) monolayer on a graphite single crystals."
R. VOLLMER, MPI, Göttingen.

"Spin dynamics in a 2-dimensional XY system."
H. ZABEL, University of Illinois.

"Multilayers of hydrogen on MgO: an interesting quantum system."
O. E. VILCHES, University of Washington, Seattle.

"Phonon densities of states of high $T_c$ copper oxides - Is electron-phonon coupling out?"
F. GOMPF and B. RENKER, KFA Karlsruhe.

"Mechanism of phase transitions in quartz."
D. ECKOLD, KFA Jülich.

# College 5

## Crystal and Magnetic Structures

"Structure-property relationships in oxide superconductors."
A. W. SLEIGHT, Central Research and Development Department, E. I. Du Pont de Nemours, Wilmington, Delaware.

"Position sensitive photomultipliers and applications."
D. SILLOU, L. A. P. P. (IN2P3-CNRS), Annecy.

"High resolution electron microscopy studies of phraseodymium oxides."
E. SCHWEDA, University of Tübingen.

"Electron density distribution of sulfur-containing compounds."
Y. WANG, National Taiwan University.

"Some Australian applications of neutron powder diffraction."
C. J. HOWARD, Department of Physics, University of Edinburgh.

"Solvent dependent structures of nickel xanthate adducts."
A. J. EDWARDS, Department of Inorganic Chemistry, University of Melbourne and Chemical Crystallography, Oxford.

"Some experiences with Rietveld analyses of $Nd_2Fe_{14}B$ hard-magnet material."
R. A. YOUNG, Georgia Institute of Technology.

"Neutron diffraction on perfect crystals."
F. EICHHORN, Zentralinstitut für Kernforschung, Dresden.

"Wide angle X-ray diffraction of single crystals and polycrystalline materials using synchrotron radiation."
J. F. BERAR, Laboratoire de Chimie-Physique du Solide, Ecole Centrale, Chatenay-Malabry.

"Practical applications of the RED extinction model."
J. KULDA, Laboratory of Neutron Physics, JINR, Dubna, USSR and Nucl. Phys. Inst., Czechoslovak Acad. of Sciences, Rez near Prague, Czechoslovakia.

"Neutron anatomy"
G. E. BACON, Sheffield University.

"A study of the intercalation of tetrahydrofuran in the $CsC_{24}$ graphitide and of the structural transition in the $Cs(THF)_xC_{24}$ intercalation compounds."
M. GOLDMANN, Centre de Recherche sur les Solides à Organisation Cristalline Imparfaite, CNRS, Orléans.

"Fe-S proteins: results of XAFS and X-ray diffraction studies."
L. C. SIEKER, University of Washington, Seattle.

"Grazing angle X-ray diffraction studies of reconstructions on semiconductor surfaces."
J. S. PEDERSEN, Risö National Laboratory.

"Magnetic structure of rare-earth multilayers."
R. ERWIN, NBS Washington.

"Complementary neutron and X-ray fibre diffraction studies of DNA."
W. FULLER, University of Keele.

# College 6

## Liquids, Disorder and Defects in Materials

"High temperature neutron diffraction studies of lattice-defect interactions in $CeO_{2-x}$."
J. FABER, Jr., Argonne National Laboratory, presently at IFF/KFA Jülich.

"Scattering from evanescent X-ray and neutron waves: application to phase transitions in semi-infinite systems."
H. DOSCH, Sektion Physik, Univ. München.

"Dynamics of first-order transition in alloys measured by small angle neutron scattering instrument."
M. FURUSAKA, Tohokn Univ., Japan, currently Rutherford Laboratory.

"TEM-in situ deformation of beryllium single crystals."
J. BEUERS, Degussa AG.

"Non-ergodic instability and the quadrupolar glass state in $K(CN)_xBr_{1-x}$ and $(NaCN)_x(KCN)_{1-x}$."
K. H. MICHEL, Department of Physics, University of Antwerp, Belgium.

"Source properties and experiments at the ESRF, in particular at high energies."
A. FREUND, ESRF Grenoble.

"Equilibrium interfaces in quasicrystals."
C. L. HENLEY, Physics Department, Boston University, Boston.

"Dynamical properties of metastable AlSi solid solutions."
J. CHEVRIER, ILL.
"Structural slowing down near the glass transition of a simple binary alloy model."
J. P. HANSEN, Ecole Normale Supérieure de Lyon.

"Spectroscopic probes for electron transfer phenomena."
R. WHITE, ILL.

"Study by inelastic neutron scattering of the vibrational modes of molecular crystals and chemisorbed molecules."
H. JOBIC, Institut de Recherches sur la Catalyse, Villeurbanne.

"Polarization analysis on liquid sodium to separate the coherent and incoherent dynamic structure factor."
O. SCHÄRPF, ILL.

# College 8 and EMBL

## Biochemistry

"The cubic phases of lipid-containing systems; structure, physical properties and biological significance."
V. LUZZATI, Centre Génétique Moléculaire, Gif-sur-Yvette.

"Enzyme construction, protein destruction and protein synthesis."
A. LILJAS, Uppsala.

"Unusual transcription regulation and usually high levels of protein expression in the late phase of vaccinia virus infection."
H. STUNNENBERG, EMBL, Heidelberg.

"Structure-function relationships in adenylate kinases."
G. SCHULZ, Albert-Ludwigs-Universität, Freiburg.

"Tubulin assembly and GTP hydrolysis."
M. J. SCHILSTRA, N. I. M. R., London.

"Three-dimensional structure and specificity of anti-lysozyme antibodies."
R. POLJAK, Institut Pasteur, Paris.

"Liquid crystalline ordering in solutions of rod-like and semi-flexible polymers including biopolymers."
H. N. W. LEKKERKERKER, Universiteit Utrecht, The Netherlands.

"Structure/function studies of calcium binding proteins: calmodulin and troponin C."
J. TREWHELLA, Los Alamos National Laboratory.

"Zinc fingers a novel protein motif for nucleic acid recognition."
A. KLUG, MRC, Cambridge.

"DNA-protein interactions at the origin of adenovirus replication."
P. VAN DER VLIET, University of Utrecht.

"Protein-nucleic acid structure and interactions in tobacco mosaic virus at 2. 9 Å resolution."
G. STUBBS, Vanderbilt University, Tennessee.

"Drug / protein interactions with the anti-tumor protein Neocarzinostatin."
L. SIEKER, University of Washington, Seattle.

"The molecular basis of virulence: a virulent mutant of rabies virus."
A. FLAMAND, Laboratoire de Génétique des Virus, Gif-sur-Yvette.

"The three-dimensional structure of a DNA duplex containing looped-out bases."
J. L. SUSSMAN, Weizmann Institute of Science, Israël.

"Enveloped virus structures."
S. FULLER, EBML Heidelberg.

"Aspects of antibiotic resistance: function and evolution."
J. DAVIES, Institut Pasteur, Paris.

"The c-myc gene in cell transformation and human cancer."
R. MONIER, I. F. S. B. M., Villejuif.

"Structure and function of an icosahedral enzyme complex: heavy riboflavin synthase from Bacillus Subtilis."
R. LADENSTEIN, Max-Planck-Institut für Biochimie, Martinsried.

"Inhibition of trypsin by chloromethyl ketone: $^{13}C$ NMR and X-ray diffraction analysis of the enzyme-inhibitor complex."
X. LEE, N. R. D., Ottawa, Canada.

"Life in the Dead Sea - Adaptation of protein structure to extreme salinity."
M. SHOHAM, Weizmann Institute, Rehovot.

"Neutron anatomy."
G. E. BACON, Sheffield University.

"Crystal packing and morphology of protein crystals."
M. FREY, CNRS, Marseille.

"Glucose isomerase: structure and function."
J. JENKINS, Orsay.

"The structure of foot and mouth disease virus."
D. STUART, Oxford.

# College 9

## Chemistry

"Small-angle neutron scattering results on bicontinuous microemulsions."
A. DE GEYER, ILL.

"The structure factor of colloidal model dispersions; the influence of colloidal forces and fluid dynamical forces."
C. G. DE KRUIF, University of Utrecht.

"Physical gelation of low molecular weight molecules in non-aqueous solvents."
P. TERECH, ILL.

"Static and dynamic light scattering from fractal colloidal aggregates."
R. KLEIN, Universität Konstanz.

"Particle motion in colloidal dispersions."
T. G. M. VAN DE VEN, University of Bristol.

"Synthesis and characterization of side chain liquid crystalline polymers with ethylene oxide spacers."
R. DURAN, MPI für Polymerforschung, Mainz.

"NMR studies of quantum tunnelling and thermally activated motions of methyl groups in molecular crystals."
H. LANG, W. W. U. Münster.

"The magnetically suspended fast chopper in the HET spectrometer on the ISIS spallation neutron source at RAL."
T. J. L. JONES, Rutherford Appleton Laboratory.

"Elastic and optoacoustic anomalies at structural transitions of polymer liquid crystals."
J. K. KRÜGER, University of Saarland, Saarbrücken.

"Investigations of micellar and microemulsion solutions using SANS."
L. MAGID, University of Tennessee, Knoxville.

"Two-dimensional melting of the nitric-acid intercalated in graphite studied by quasi-elastic neutron scattering."
C. SIMON, Université Paris VII.

"Diffusion of linear and star polymers in concentrated solutions."
T. P. LODGE, University of Minnesota, Minneapolis.

"Source properties and experiments at the ESRF, in particular at high energies."
A. FREUND, ESRF, Grenoble.

"Staging in layer intercalates and doped polymers."
J. I. FISCHER, University of Pennsylvania and Montpellier.

"Experimental facilities at ESRF, in particular in small-angle scattering."
C. RIEKEL, ESRF Grenoble.

"Crossover from Rouse to reptation: a molecular dynamics simulation."
K. KREMER, Institut für Physik, Universität Mainz.

"Optical spectroscopy studies of phase situation and molecular motion in some liquid crystals."
W. WITKO, Institute of Nuclear Physics, Krakov.

"Studies of association in compound-forming and glass-forming liquids."
D. QUITMANN, F. U. Berlin.

"New developments in elastic light scattering."
O. GLATTER, Inst. f. Physikalische Chemie, Universität Graz.

"Are microemulsions spherical droplets?"
T. ZEMB, DPC, Saclay.

# Workshop Proceedings, Books and Theses

## Workshops Organized in 1988:

DIANOUX A.J., PETRY W., RICHTER D., TEIXEIRA J.:
ILL Workshop on Dynamics of Disordered Materials, ILL, Grenoble, September 26-28, 1988. (Abstracts, 88DI24T)

GLAESER W., ROSSAT-MIGNOD J., SCHWEIZER J., REGNAULT L.P., MAIER B., VETTIER C.:
ICNS'88. International Conference on "Neutron Scattering", Grenoble, July 12-15, 1988. (Programme and abstracts, 88GL28T)

C. JANOT, J.M. DUBOIS E: Quasicrystalline materials. ILL/CODEST Workshop, Grenoble, March 21-25, 1988

## Sponsored by the ILL:

UNIV. BARCELONA, ILL :
II Curso Difusion de Neutrones, Centre d'Estudis Avançats, CSIC, BLANES, SPAIN, 14-18 NOVIEMBRE 1988 (CSIC, 1988)

## Workshops Published in 1988:

Polymer motion in dense systems, Proceedings of the Workshop Grenoble, France, September 23-25, 1987, D. Richter, T. Springer. Eds., Springer Proceedings in Physics, Vol. 29 (Springer-Verlag, 1988) (pp.310)

Quasicrystalline materials. Proceedings of the ILL/CODEST Workshop, Grenoble, March 21-25, 1988, C. JANOT, J.M. DUBOIS EDS.
(WORLD SCIENTIFIC, 1988) (pp. 434)

## Books

BARBARA B., GIGNOUX D., VETTIER C.:
Lectures on modern magnetism.
(SCIENCE PRESS AND SPRINGER-VERLAG, 1988)
BEE M. :
Quasielastic neutron scattering. Principles and applications in solid state chemistry, biology and materials science.
(A. HILGER, 1988)

## Translations

KRAXENBERGER H.:
Construction and application of a curved crystal in neutron scattering. Thesis, University of Bayreuth.
TRANSLATED FROM GERMAN BY D.R.GRAY.

LOEHNEYSEN L.VON:
Numerical studies on the renormalisation of the O4 theory on the lattice.
TRANSLATED FROM GERMAN BY D.R. GRAY

## Theses

AKER E.:
Untersuchungen zur neutroneninduzierten Spaltung des Californium-Isotops $Cf^{249}$.
DISSERTATION, UNIVERSITAET KARLSRUHE, FEBRUAR 1987

BAUMANN J.:
Experimentelle Grenze fuer die elektrische Ladung des freien Neutrons.
DISSERTATION, UNIVERSITAET BAYREUTH, MAI 1988

BRUECKEL T.:
Magnetische Fehlordnung in dem Mischgranatsystem $(Fe_xCr_{1-x})_2Ca_3(GeO_4)_3$.
DISSERTATION, FAKULTAET FUER PHYSIK DER EBERHARD-KARLS-UNIVERSITAET ZU TUEBINGEN, 1988

DEGENHARDT D.:
Struktur und Phasenübergänge von adsorbierten Monolagen auf MgO-Pulver. (Untersucht mit Röntgen- und Neutronenstreuung)
DISSERTATION, UNIVERSITAET KIEL, 1988

FOERSTER I.:
Die Kernstruktur des $^{191}Os$ und seine Interpretation im Rahmen des Nilsson- und des Interacting Boson-Fermion Modells.
DISSERTATION, UNIVERSITAET KOELN, 1988

GALEZ P.:
Contribution à l'élaboration de monochromateurs en graphite pyrolytique.
THESE, UNIVERSITE JOSEPH FOURIER, GRENOBLE I, DECEMBRE 1988.

GOLDMANN M.:
Etude, par diffusion neutronique, de l'insertion du tétrahydrofuranne dans le graphiture $CsC_{24}$ et des transitions structurales dans les composés intercalaires $Cs(THF)_xC_{24}$.
THESE, UNIVERSITE PARIS 7, JUIN 1988

LACORRE P.:
La frustration magnétique: quelques manifestations expérimentales, leur simulation et une approche théorique.
THESE, UNIVERSITE DU MAINE, DECEMBRE 1988.

MAACK A.:
Praxissemester-Bericht.
PRAXISSEMESTER-BERICHT 1988/09/01-1988/01/31

SCHILDBERG H.P.:
Neutron diffraction studies of the two-dimensional quantum gases $^3He$, $^4He$ and D2 adsorbed on graphite.
DISSERTATION, MATHEMATISCH-NATURWISSENSCHAFTLICHEN FAKULTAET DER CHRISTIAN-ALBRECHTS-UNIVERSITAET ZU KIEL, 1988

SCHOOLMANN D.:
Die Nebelkammer und die Darstellung von gasförming/flüssigen Kolloiden.
PRAXIS-SEMESTERBERICHT 1987/09/01-1099/01/31

This list groups all the known publications resulting from the research at the I.L.L. during 1988.
ILL Reports are listed first (number 1 to 99). They are followed by the list of publications in the scientific literature (journals, conference proceedings, books) with ILL authors and coauthors (100 to 999) and by publications related to experimental work performed at the ILL but without ILL author or coauthor (1000 to 1100).

# Internal Reports

## (G = General Reports, T = Technical Reports)

**88LA1T**
LAMOREAUX S.K.:
Deuterated polystyrene-Synthesis and uses for ultracold neutron bottles and the neutron EDM experiment.

**88RO2T**
ROSTA L., ZSIGMOND GY., FARAGO B., MEZEI F., BAN K., PERENDI J.:
Multidisc neutron velocity selector. (KFKI-1987-79/E)

**88CA3G**
CASTETS C. [Ed.]:
1987 ILL Experimental Reports and Theory College Activities.

**88BR4T**
BROWN P.J.:
Tables of magnetic form factors. (Section for inclusion in Vol. C of the new edition of the International Tables of Crystallography)

**88FI5T**
FILHOL A., BLANC J.Y., ANTONIADIS A., BERRUYER J.:
ABFfit for the Vax. A maximum likehood profile refinement program for poissonnian patterns. Manual for the version 3.0

**88GU6T**
GUILLAUME F.:
Analysis of "tof" data on DIVA.

**88G7T**
GOPPELT P., BLANC Y., MAGERL A.:
Stress test of pyrolytic graphite.

**88BA8T**
BARUCHEL J.:
Imagerie avec le rayonnement synchrotron (radiographie, topographie, théorie dynamique).
SESSION D'ETUDES "LE RAYONNEMENT SYNCHROTRON", Grenoble, 25-29 Avril 1988

**88SC9T**
SCHRECKENBACH K.:
Possible time reversal experiment with polarized cold neutrons.

**88FE10T**
FERNANDEZ-DIAZ M.T:
Quasi-elastic neutron scattering from $YBa_2Cu_3O_{7-x}$ compounds.

**88CO11T**
COLVIN G.G., HOYLER F., ROBINSON S.J.:
Gamma rays from the reaction $^{99}Ru(n,\gamma)$

**88TH12T**
THOMAS M.:
Comportement d'une membrane circulaire, plane, encastrée et précontrainte soumise à une pression uniformément répartie (Rapport et Additif).

**88LA13T**
LACORRE P.:
MCMAG. Manual. Version 88.01

**88ST14T**
STANSFIELD R.F.D., WILKINSON C.:
Neutron area diffractometry and spatial deconvolution techniques.
TECHNICAL REPORT. "COMPUTATIONAL ASPECTS OF PROTEIN CRYSTAL DATA ANALYSIS". PROCEEDINGS DARESBURY STUDY WEEKEND, SERC 1987, DL/SCI/R25

**88CH15T**
CHAHID A.:
Rapport de stage effectué à l'Institut Laue-Langevin, Juin-juillet 1987.

**88JA16T**
JANOT C., DUBOIS J.M.:
ILL/CODEST Workshop on quasicrystalline materials, Grenoble, March 21-25, 1988. Abstracts.

**88LE17G**
LEHMANN M.S.:
Structure analysis from powder patterns.
LECTURE GIVEN AT NORDIC COURSE ON SYNCHROTRON RADIATION, AARHUS AND SANDBJERG, AUGUST 19-26, 1988.

**88KU18T**
KUEST H.:
Program WLK and for data preparation: Programs INTER, BS-WLK and TOF-WLK.

**88FO19T**
FOUBERT J.:
Programmes de contrôle de l'instrument IN6 sur VAXstation II/GPX.

**88KU20T**
KUEST H.:
Program DEORIE. Modifications.

**88NU21T**
NUTLEY M.:
Investigation into compounds containing mixed valency states and layered oxide structures of bismuth and thallium.

**88GO22T**
GOLUB R., GAEHLER R.:
Spin 1/2 particles with $<\sigma>$ 100, "weak value" of a quantum observable and "polarization amplification".

**88AN23T**
ANTONIADIS A., FILHOL A.:
TEX on VAX and on Macintosh.

**88DI24T**
DIANOUX A.J., PETRY W., RICHTER D., TEIXEIRA J.:
ILL Workshop on Dynamics of Disordered Materials, Grenoble, September 26-28, 1988. (Abstracts)

**88TA25G**
TASSET F., MATHIEU B.:
Instrument D3B: Procédure d'échange pour les cassettes monochromateur.

**88GH26T**
GHOSH R.:
Principles of operation of the computer control systems on D11 and D17 small angle neutron spectrometers at the Institut Laue-Langevin.

**88DE27G**
GEYER A. DE, TERECH P.:
Short computing guide for use of ILL visitors. Small angle scattering data.

**88GL28T**
GLAESER W., ROSSAT-MIGNOD J., SCHWEIZER J., REGNAULT L.P.,
MAIER B., VETTIER C.:
ICNS'88. International Conference, Grenoble (France), July 12-15, 1988. Programme and abstracts.

**88MA29T**
MARTINEZ J.L.:
Introducción a las tecnicas experimentales con neutrones.
II CURSO DIFUSION DE NEUTRONES, BLANES, SPAIN, NOVIEMBRE 1988, CAPITULO 2, 1-14

**88RO30T**
RODRIGUEZ-CARVAJAL J., PANNETIER J.:
Chemical crystallography with neutrons. I. Fundamentals of neutron diffraction.
II CURSO DIFUSION DE NEUTRONES, BLANES, SPAIN, NOVIEMBRE 1988, CAPITULO 3.1, 1-19

**88RO31T**
PANNETIER J., RODRIGUEZ-CARVAJAL J.:
Chemical crystallography with neutrons. II. Applications of neutron powder diffraction.
II CURSO DIFUSION DE NEUTRONES, BLANES, SPAIN, NOVIEMBRE 1988, CAPITULO 3.2, 1-34

**88JA32T**
JANOT C.:
An introduction to small-angle neutron scattering: some application to materials science.
II CURSO DIFUSION DE NEUTRONES, BLANES, SPAIN, NOVIEMBRE 1988, CAPITULO 4, 1-35

**88DU33T**
DUBOIS J.M., JANOT C.:
Amorphous alloys and quasicrystals studied by neutron diffraction.
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**88DA34T**
DAUVERGNE B.:
Notice de l'Instrument D11.

**88HE35T**
HEIDEMANN A.:
Problems of physical chemistry studied by high resolution incoherent neutron scattering.
II CURSO DIFUSION DE NEUTRONES, BLANES, SPAIN, NOVIEMBRE 1988, CAPITULO 11, 1-25

**88RI36T**
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**88BR37T**
BROWN P.J.:
Introduction to polarized neutrons.
II CURSO DIFUSION DE NEUTRONES, BLANES, SPAIN, NOVIEMBRE 1988, CAPITULO 16, 1-10

**88DO38T**
DORNER B.:
Introduction to inelastic neutron scattering.
II CURSO DIFUSION DE NEUTRONES, BLANES, SPAIN, NOVIEMBRE 1988, CAPITULO 17, 1-10

**88MA39T**
MARTINEZ J.L.:
Estudio de excitaciones magneticas mediante difusion inelastica de neutrones.
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**88DO40T**
DORNER B.:
Collective excitations measured with inelastic neutron scattering and with inelastic x-ray scattering.
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**88VE41T**
VETTIER C.:
Neutron scattering studies on some high $T_c$ compounds and heavy fermion systems.
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**88BR42T**
BROWN P.J.:
Spin densities in solids and molecules.
II CURSO DIFUSION DE NEUTRONES, BLANES, SPAIN, NOVIEMBRE 1988, CAPITULO 21, 1-10

**88VE43T**
VETTIER C.:
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**88RE101**
RENAUDIN J., FEREY G., KOZAK A. DE, SAMOUEL M., LACORRE P.:
Crystal and magnetic structures of the ferrimagnet $Ba_2Ni_7F_{18}$.
SOLID STATE COMMUNICATIONS **65**, 185-188 (1988)

**88EC102**
ECKERT J., KUBAS G.J., DIANOUX A.J.:
Rotational tunneling of bound $H_2$ in a tungsten complex.
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**88UR103**
URBAN R., EHRHART P., SCHILLING W., SCHOBER H.R., LAUTER H.J.:
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**88NI104**
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**88OE105**
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**88LO106**
LOIDL A., KNORR K., ROWE J.M., McINTYRE G.J.:
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**88CH107**
CHATTOPADHYAY T., BROWN P.J., THALMEIER P., BAUHOFER W.,
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**88RE108**
RENKER B., GOMPF F., GERING E., FRINGS P.H., RIETSCHEL H., FELTEN R., STEGLICH F., WEBER G.:
Normal ground-state properties of heavy-fermion actinide compounds.
PHYSICA B **148**, 41-44 (1987)

**88TU109**
TUN Z., NELMES R.J., KUHS W.F., STANSFIELD R.F.D.:
A high-resolution neutron-diffraction study of the effects of deuteration on the crystal structure of $KH_2PO_4$.
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**88SU110**
SUBBER A.R.H., HAMILTON W.D., VAN ISACKER P., SCHRECKENBACH K., COLVIN G.G.:
Monopole transitions in $^{172}Yb$ and the nature of the 1405 keV $O^+$ level.
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**88DU111**
DUBOIS J.M., JANOT C.:
Chemical isomorphism and partial pair distribution functions in decagonal Al-Mn.
EUROPHYSICS LETTERS **5**, 235-240 (1988)

**88JA112**
JAMES S.D., PEARCE J.A., OLIVER R.A.:
The geochemistry of the lower proterozoic Willyama Complex volcanics, Broken Hill Block, New South Wales.
IN "GEOCHEMISTRY AND MINERALIZATION OF PROTEROZOIC VOLCANIC SUITES", T.C. PHARAOH ET AL., EDS., GEOLOGICAL SOCIETY, SPECIAL PUBLICATION N° 33, 395-408.

**88SU113**
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Neutron diffraction of quasiperiodic structures: contrast variation, isomorphism and pseudo-symmetries.
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**88FR116**
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Study of the glass transition order parameter in amorphous polybutadiene by incoherent neutron scattering.
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**88GU117**
GUILLAUME F., YARWOOD J., PRICE A.H.:
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**88CH120**
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The tensor of compositional deformation. A new crystallographic way to analyse syncrystallization.
JOURNAL OF APPLIED CRYSTALLOGRAPHY **21**, 10-14 (1988)

**88BA121**
BARUCHEL J., KURODA K., LIAUD P., MICHALOWICZ A., SILLOU D.:
A position-sensitive detector for neutron diffraction topography.
JOURNAL OF APPLIED CRYSTALLOGRAPHY **21**, 28-32 (1988)

**88ST122**
STEYERL A., DREXEL W., EBISAWA T., GUTSMIEDL E., STEINHAUSER K.A., GAEHLER R., MAMPE W., AGERON P.:
Neutron microscopy.
REVUE DE PHYSIQUE APPLIQUEE **23**, 171-180 (1988)

**88MA123**
MARCUS B., TOUZAIN P., SOUBEYROUX J.L.:
Kinetic study of electrochemical intercalation of potassium solvated by tetrahydrofuran into graphite.
SYNTHETIC METALS **23**, 13-17 (1988)
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**88BA124**
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Critical behavior of the diffusion constant in the melting of the layer of nitric acid intercalated in graphite.
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**88GO125**
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**88BE128**
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Melting of the intercalated layers in $Cs_x(THF)_yC_{24}$.
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LOPEZ-CABARCOS E., GONZALEZ-ARCHE A., BATALLAN F., FRICK B.: Structure and dynamics of vinylidenefluoride-trifluoroethylene copolymers. SPRINGER PROCEEDINGS IN PHYSICS (5TH IFF-ILL WORKSHOP ON "MOLECULAR BASIS OF POLYMER NETWORKS", JUELICH, OCT. 88). (88LO5242)

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OLBRICH E., CHEN D., LINDNER P., ZACHMANN H., BENOIT H.: Chain conformations and average degree of polymerization of the sequences in copolyesters. PHYSICA B (PROCEEDINGS OF THE ICNS'88, GRENOBLE, JULY 12-15, 1988). (88OL5156)

RICHTER D., FARAGO B., EWEN B., OESER R.: The fluctuations of cross-links in a rubber - a neutron spin echo study. PHYSICA B (PROCEEDINGS OF THE ICNS'88, GRENOBLE, JULY 12-15, 1988). (88RI5070)

RICHTER D., FARAGO B., FETTERS L., HUANG J.S., EWEN B.: Neutron spin echo investigation on the dynamics of star polymers. SPRINGER PROCEEDINGS IN PHYSICS (5TH IFF-ILL WORKSHOP ON 'MOLECULAR BASIS OF POLYMER NETWORKS', JUELICH, OCT. 1988). (88RI5190)

RICHTER D., FARAGO B., HUANG J.S., FETTERS L.J., EWEN B.: A study of single arm relaxation in a polystyrene star polymer by neutron spin echo spectroscopy. MACROMOLECULES. (88RI5002)

ROSSLE W., LINDNER P., DETTENMAIER M.: Deformation of glassy polycarbonate studied by neutron scattering. PHYSICA B (PROCEEDINGS OF THE ICNS'88, GRENOBLE, JULY 12-15, 1988). (88RO5157)

TEITGE A., CHEN D., KUGLER J., LINDNER P., ZACHMANN H.: Chain conformations in oriented poly(ethylene terephthalate). PHYSICA B (PROCEEDINGS OF THE ICNS'88, GRENOBLE, JULY 12-15, 1988). (88TE5155)

TERECH P.: Orientation of rods formed by aggregated surfactants in organic media: the steroid-cyclohexane physical gel case. PROCEEDINGS OF THE 4TH EPS CONFERENCE ON THE "HYDRODYNAMICS OF DISPERSED MEDIA", ARCACHON, MAY 24-27, 1988. (88TE5163)

TERECH P.: Orientation of rod-like surfactant aggregates in a non aqueous physical gel. PROGRESS IN COLLOID & AND POLYMER SCIENCE (PROCEEDINGS OF THE 2ND ECIS CONFERENCE, ARCACHON, SEPT. 19-22, 1988). (88TE5064)

WICKER A., BERGE B., LAJZEROWICZ J., LEGRAND J.F.: Non-linear optical investigation of the bulk ferroelectric polarization in thin films of VF2-TrFE copolymers. FERROELECTRICS (PROCEEDINGS OF THE 1ST ECAPD & ISAF 88, ZÜRICH, AUG. 29-SEPT. 1, 1988). (88WI5165)

The following is the list of experimental reports performed at the I.L.L. up to the end of 1988 for which experimental reports were received before January 17th 1989.
For a detailed study of the results, please refer to the volume "1988 EXPERIMENTAL REPORTS AND THEORY COLLEGE ACTIVITIES"
(Reports marked with * are not available for publication)

# College 3

## Fundamental and Nuclear Physics

**3-01-220, 3-01-229** **PN1**
Mass and nuclear charge distribution of fission fragments from $^{241}Am(2n_{th},f)$.
SCHUNK T., SIEGLER P., MUTTERER M., THEOBALD J.P.

**3-01-221, 3-01-230** **PN1**
Fission product yields in the very asymmetric splits of $^{239}Pu(n_{th},f)$.
DITZ W., DENSCHLAG H. V., FAUST H.

**3-01-223** **PN1**
Energy loss and energy straggling of fission fragments in thin foils. GELTENBORT P., GOENNENWEIN F., GRAF U., LOEFFLER H., PAUWELS J., BOERSIG B., AIT-SALEM M.

**3-01-224, 3-01-239, 3-01-24** **PN1**
Search for thermal neutron induced heavy ion emission from heavy nuclei.
BOERSIG B., GELTENBORT P., GOENNENWEIN F., LOEFFLER H.

**3-01-228** **PN1**
Emission of light charged particles accompanying thermal neutron induced fission of $^{235}U$.
MUTTERER M., THEOBALD J. P., BARREAU G., LEROUX B., BAUM W., ZOELLER C., DOAN T. P., SICRE A.

**3-01-229** see **3-01-220**
**3-01-230** see **3-01-221**

**3-01-230** **PN1**
Fission product yields in the very asymmetric mass split of $^{239}Pu(n_{th},f)$.
DENSCHLAG H.O., DITZ W.

**3-01-231** **PN1**
Nuclear charge distribution of fission fragments in $^{229}Th(n,f)$.
WILHELMY J., FOWLER M., DJEBARA M., ASGHAR M., BOCQUET J.P., BRISSOT R.

**3-01-233** **PN1**
Qβ-measurements of light fission products in the vicinity of the main neutron shell number N = 50.
MUENNICH F., KEYSER U., BALOG K., GRAEFENSTEDT M., OTTO T., SCHREIBER F., WULFF J.

**3-01-234** **PN1**
Emission of light charged particles accompanying thermal neutron induced fission of $^{235}U$.
DOAN T.P., BAUM W., BARREAU G., GOEPFERT A., LEROUX B., MUTTERER M., SICRE A., THEOBALD J.P.

**3-01-23** see **3-01-241**
**3-01-24** see **3-01-224**

**3-01-246** **PN1**
Measurement of the β–decay half-lives of some new isotopes of Ni and Cu.
ARMBRUSTER P., BERNAS M., BOCQUET J.P., BRISSOT R., FAUST H.R., KOZHUHAROV C., SIDA J.L.

**3-01-248** **PN1**
Total β-decay energy measurements of rare earth nuclides with mass numbers A ≥ 150.
MUENNICH F., KEYSER U., BALOG K., GRAEFENSTEDT M., JUERGENS P., SCHREIBER F., WINKELMANN T.

***3-02-352, 3-02-389,** **PN2**
Identification of EO transition in $^{202}Hg$ and in $^{198}Os$ $^{190}0s$.
HAMILTON W.D.

***3-02-362** **PN3**
Resonant scattering and absorption of gamma-radiation in $^{114}Cd$ and $^{200}Hg$.
HAMILTON W.D.

**3-02-364** **IN4**
Thermal neutron capture in $^{31}P$.
MICHAELSEN S., LIEB K.P., WINTER C., VON EGIDY T.

**3-02-365** **PN3/PN4**
Precise atomic mass determination of the $^{76}Ge$ nucleus and its relation to the double β decay.
BARREAU G., HUBERT P., LECCIA F., MENNRATH P.

**3-02-368** **PN2**
Calibration of a plastic-scintillator telescope for high-energy electrons.
MUENNICH F., KEYSER U., SCHREIBER F., WINKELMANN T.

***3-02-370** **PN2**
Decays of 1+ mixed symmetry state in $^{158}Gd$. Are they predominantly M1?
HAMILTON W.D.

***3-02-371** **PN2**
$^{180}Hf$. The internal conversion of transitions and their order and type.
HAMILTON W.D.

**3-02-374** **PN2**
Search for a resonance in the excitation function of $e^+e^-$ scattering.
KRUSCHE B., SCHRECKENBACH K., KOZHUHAROV C., TSERTOS H.

**3-02-377** **PN3, PN2**
A study of the reaction $^{103}Rh(n,\gamma)^{104}Rh$.
KRUSCHE B., MICHAELSEN S, TUKROURI F., MCMAHON T.

**3-02-379** **PN3, PN4**
Velocity dependence of stopping cross sections of lithium projectiles.
HAUSER U., NEUWIRTH W., HATCH E.

**3-02-382** **GAMS4**
Absolute gamma spectroscopy at high energies using a two axis flat crystal spectrometer (GAMS4).
DEWEY M.S., GREENE G.L., KESSLER E.G., DESLATTES R.D.

**3-02-383** **PN3**
Test of U(5) or O(6) character in $^{196}Pt$ by lifetime measurements via GRID.
CASTEN R.F., BOERNER H.G., JOLIE J., ROBINSON S., SCHILLEBEECKX P.

**3-02-384** **PN3**
Stopping power of 0.5 keV Fe and Ti atoms measured with GAMS4.
ULBIG S., LIEB K.P., WINTER C., BOERNER H.G., JOLIE J., ROBINSON S.

**3-02-385** **GAMS4**
Lifetime measurements in $^{54}Cr$ by means of GAMS4.
LIEB K.P., WINTER C., ULBIG S., BOERNER H.G., JOLIE J., ROBINSON S., DEWEY M.S.

**3-02-389 see 3-02-352**

**3-02-402** **PN3**
Study of the $^{191}Ir(n,\gamma)$ reaction.
DRISSI S., KERN J., BOERNER H.G., ROBINSON S., SCHILLEBEECK P.

**3-05-X** **H17**
Interaction of ultra-cold neutrons with superfluid helium and a superthermal source of UCN.
GOLUB R., MEZEI F., BUTTERWORTH J.

**3-06- 78** **PN6**
Shape coexistence in the N=59 isotone $^{97}Sr$.
LHERSONNEAU G., PFEIFFER B., KRATZ K.L., OHM H., SISTEMICH K.

**3-06- 94** **SN6**
High-resolution time-of-flight spectroscopy of beta-delayed neutrons.
WOEHR A., PFEIFFER B., LEIST B., KRATZ K.L., CRAWFORD G.I, STOHLKER U.

**3-07- 25, 3-13-123** **SN7, S34**
Measurement of $\eta$ $^{235}U$ for sub-thermal neutrons.
WARTENA J.A., WEIGMANN H., GELTENBORT P., SCHRECKENBACH K., BUERKHOLZ C., KECK B.

**3-07- 29** **SN7**
Parity on cold fission.
GOENNENWEIN F., GRAF U., GELTENBORT P., SCHRECKENBACH K., OED A.

**3-07- 31** **SN7**
Prototype of a zero-fiel neutron spin echo spectrometer.
DUBBERS D., EL.MUZEINI P., KESSLER M., LAST J.

**3-08- 6** **PN8**
Ternary fission of $^{246}C$.
THEOBALD J.P., LEROUX B., KOCZON P., MUTTERER M., BARREAU G.

**3-08- 6** **PN8**
Mass- and energy distribution of fission fragments from the Cm(n,f) reaction.
THEOBALD J.P., MOORE M.S., KOCZON P., GELTENBORT P., OED A.,PANNICKE J., MUTTERER M.

**3-08- 25** **PN8**
Multiparametric fission study with Cosi Fan Tutte of $^{239}Pu$.
ASGHAR M., BOUCHENEB M., BARREAU G., DOAN T.P., LEROUX B., SICRE A., GOENNENWEIN F., OED A., AKER E., ENGELHARDT D.

**3-08- 26** **PN8**
Hot fragmentation in $^{235}U(n_{th},f)$.
MOLLENKOPF W., GOENNENWEIN F., GELTENBORT P., OED A., GRAF U., BOERSIG B., LOEFFLER H.

**3-08- 27, 3-08- 31** **PN8**
Nuclear charges, mass and energy of fragments in $^{249}Cf(n_{th},f)$.
AKER E., ENGELHARDT D., SCHWEE C., GELTENBORT P., GOENNENWEIN F., WILKINS B.

**3-08- 29** **PN 8**
A detailed investigation of the $^{229}Th(n_{th},f)$ reaction with Cosi Fan Tutte.
BOUCHENEB N., GELTENBORT P., ASGHAR M., BARREAU G., DOAN T.P., GOENNENWEIN F., LEROUX B., OED A., SICRE A.

**3-08- 31 see 3-08- 27**

**3-13-103** **H22F**
The character of the 1330 keV level in $^{111}Cd$ and $(n,\gamma)$ studies of $^{111}Cd$.
HAMILTON W.D.

**3-13-104** **SP.BE**
Gamma-gamma directional correlations in $^{200}Hg$ and the identification of $1^+$ mixed-symmetry state.
HAMILTON W.D.

**3-13-108** **H22E,H**
Neutron capture radiography of Li, B and N in plant and animal systems
THELLIER M., WISSOCQ J.C., FERNANDEZ T., MARTINI F., LAURENT-PETTERSSON M., OUZNADJI M., FOURCY A.

***3-13-111** **H22E,F**
Spin assignments in Hf and its band structure.
HAMILTON W.D.

**3-13-114** **H22D**
Systematic study of $(N,\alpha)$ and (N,F) reaction.
WAGEMANS C., SCHILLEBEECKX P., BOCQUET J. P

**3-13-123** **H22E,F**
Measurement of η of $^{235}U$ for subthermal neutron energies.
KECK B., WARTENA J.A., WEIGMANN H., BUERKHOLZ C.

**3-13-123** **see** **3-07- 25**

***3-14-** **SP.BE**
Acceleration (deceleration) of neutrons in time-dependent magnetic field.
NIEL L., RAUCH H.

**3-14-TU** **Neutron Turbine**
Use of mirrors and multilayers in cold and ultracold neutron optics.
STEYERL A., NAGEL H., DREXEL W., MALIK S.S., GUTSMIEDL E.,TURBA W.

**3-15- 4** **S50**
Determination of h/m.
KRUEGER E., NISTLER W., WEIRAUCH W.

# College 4

## Structural and Magnetic Excitations

**4-01-323** **IN8**
Phonons in malonic acid.
MONKENBUSCH M., GRIMM H.

**4-01-330** **IN4**
Temperature dependence of the phonon density of states of non-equilibrium Al-Si solid solutions.
CHEVRIER J., SUCK J.B., JANOT C., CAPPONI J. J.

**4-01-332** **IN4**
Phonon spectrum of $YBa(Cu_{1-x}Me_x)O_{7-d}$.
GOMPF F., RENKER B., REICHARDT W.

**4-01-336** **IN8**
Phonon anomalies in the high temperature phase of β-Ti and β-Zr.
PETRY W., HEIMING A., TRAMPENAU J., VOGL G.

**4-01-342** **IN6**
$YBa_2Cu_3O_{7-x}$.
RENKER B., GERING E.

**4-01-343** **IN6**
Density of phonon states in hydrogenated and deuterated acetanilide.
BARTHES M., SAUVAJOL J.L., ALMAIRAC R., MORET J.

**4-01-344** **IN12**
Solitons in deuterated acetanilide coupling with acoustic phonons. BARTHES M., ALMAIRAC R., SAUVAJOL J.

**4-01-349** **IN6**
Phonon density of states of the quasi-one dimensional superconductor $Tl_2Mo_6Se_6$.
BRUSETTI R., BONJOUR E., GOUGEON P.

**4-02-228** **IN11**
Dynamics of the incommensurate phase transition of $NaNO_2$.
DURAND D., BERNARD L., CURRAT R., MEZEI F.

**4-02-238** **D10**
Ordering in $Cu_3Pd$.
SCHWEIKA W., VETTIER C.

**4-02-240** **IN8**
Acoustic modes in the icosahedral phase $Al_6Li_3Cu$.
JANOT C., BOISSIEU M. DE , CURRAT R., DUBOIS J.M.

**4-03-410** **IN20**
Polarised neutron study of the commensurate excitation of chromium.
STIRLING W.G., PYNN R., MCEWEN K.A., LINDLEY E.J.

**4-03-414** **IN12**
Low energy excitations in $CeB_6$.
REGNAULT L.P., ERKELENS W.A.C., ROSSAT-MIGNOD J.

**4-03-440** **see** **4-03-493**

**4-03-454, 4-03-485** **IN12**
Spin wave dynamics in the garnet $Fe_2Ca_3(GeO_4)_3$ with degenerated antiferromagnetic ground state.
BRUECKEL T., PRANDL W., DORNER B., PLAKHTY V.P., GUKASOV A.G., SMIRNOV O.P.

**4-03-455** **IN3**
Antiferromagnets with dynamically interacting sublattices. Spin waves in the garnet $Mn_3Al_2Ge_3O_{12}$.
BRUECKEL T., DORNER B., GUKASOV A., PLAKHTY V., PRANDL W., SMIRNOV O.

**4-03-458** **IN5**
Hyperfine splitting of crystal field transitions in holmium ethylsulphate.
CARBONI C., MCMORROW.D., MITCHELL P.W.

**4-03-461** **IN8**
Magnetic excitations in $CsFeBr_3$, a 1-D antiferromagnet with very large planar anisotropy.
STEINER M., KAKURAI K., VISSER D., DORNER B.

**4-03-464** **IN20, IN8**
Magnetic fluctuations and excitations in the s=1 AF chain NENP.
REGNAULT L.P., RENARD J.P., VERDAGUER M.

**4-03-469** **D7**
Wavenumber dependent susceptibility of the heavy fermion metal $CeCu_6$.
MEZEI F., FISK Z., SMITH J.L., SEVERING A., PAPPAS C.

**4-03-476** **IN4**
Crystal field in rare earth disilicides.
GALERA R.M., PIERRE J.

**4-03-478** **IN4**
Inelastic spectrum of the heavy fermion $CeInCu_2$.
NAJIB A., PIERRE J.

**4-03-485** see **4-03-454**

**4-03-489** **IN8**
Magnetic excitations in $CoCl_2$-GIC of stage 1.
ROGERIE J., SIMON C.

**4-03-493, 4-03-440** **IN4, IN6**
Crystal field excitations in $CeD_{2+x}$.
ARONS R.R., ABELN A., MUTKA H.

**4-03-497** **IN4**
Crystal electric fields (CEF) in $REP_5$.
LOEWENHAUPT M., HOFFMANN P., CHATTOPADHYAY T.

**4-03-499** **IN5, IN10**
Direct observation of anisotropic exchange interactions in f-electron systems.
FURRER A., GUEDEL H.U.

**4-03-500** **IN20**
Nature of magnetic excitations in $CeSi_{1.7}$.
HIPPERT F., REGNAULT L.P., ROSSAT-MIGNOD J., KOHGI.

**4-03-504** **IN8**
Study of magnetic excitations in single-crystal monodomain UTe.
LANDER G.H., STIRLING W.G., HAGEN M., ROSSAT-MIGNOD J.

**4-03-507** **IN20**
Study of spinwave linewidth in 1-D ferromagnetic system using polarization analysis.
KAKURAI K., STEINER M., PYNN R., REITER G., DORNER B.

**4-03-510** **IN20**
Thermally induced longitudinal spin fluctuations in TMMC due to spin-energy coupling.
BOUCHER J.P., REGNAULT L.P., PYNN R.

**4-03-512** **IN20**
Study of spin dynamics in the spin-flop phase and search for nonlinear excitations in the 2-D-AFM $MnTiO_3$.
STEINER M., KAKURAI K., PYNN R.

**4-03-513** **IN11**
Nonlinear excitations in the 2D-planar magnet $BaNi_2(PO_4)_2$.
REGNAULT L.P.

**4-03-514** **IN1**
Measurement of high energy magnetic excitations in hexagonal cobalt by inelastic neutron scattering.
SQUIRES G.L., PERRING T.G., HAYDEN S.M.

**4-03-518** **IN12**
The antiferromagnets with dynamically interacting sublattices. Spin waves in the garnet $Cr_2Ca_3(GeO_4)_3$.
BRUECKEL T., DORNER B., PLAKHTY V.P., CHERNENKOV Y., PRANDL W.

**4-03-525** **IN8**
Investigation of spin waves along selected high symmetry directions in $Nd_2Fe_{14}B$ and $Y_2Fe_{14}B$.
STEINER M., MAYER H.M., DORNER B., SCHMIDT B., WIETHOFF P.

**4-03-529** **IN12**
Magnetic excitations and spin fluctuations in CeSe.
DOENNI A., FURRER A., HAYDEN S.

**4-03-532** **IN1**
High energy magnetic excitations in $La_2Cu0_4$.
AEPPLI G., BROHOLM C., FISK Z., HAYDEN S.M., MOOK H.

**4-03-533** **IN8**
Magnetic excitation in $YBa_2Cu_30_{6+x}$.
ROSSAT-MIGNOD J., REGNAULT L. P., JURGENS M.J., BURLET P., VETTIER C.

**4-04-165** **IN12**
Spin waves in the Heisenberg ferromagnet, europium oxide, near to, but below $T_c$.
MITCHELL P.W., LLOYD R.G., MOOK H.A.

**4-04-166** **IN11, IN12**
Possible lineshape cross-over in the dynamics of critical fluctuations at the Curie point in EuO.
MEZEI F., STIRLING W.G.

**4-04-169** **IN12**
Longitudinal susceptibility of $RbMnF_3$.
COWLEY R.A., BATES S., COX U.J.

**4-04-173** **IN1**
Magnetic excitations of the spin density wave in chromium.
MITCHELL P.W., BOOTH J.G, STIRLING W.G., PAUL D. MCK., HAYDEN S.M.

**4-04-176** **IN12**
Spin dynamics in the dipolar Ising ferromagnet $Li(Tb_{0.6}Y_{0.4})F_4$.
MITCHELL P.W., LLOYD R.G.

**4-04-177** **IN20**
High-resolution, polarised neutron study of the ordered ferromagnetic compound $Pt_3Mn$.
PAUL D. MCK., STIRLING W.G., BOOTHROYD A.T.

**4-04-178** **IN12**
Critical dynamics in uranium nitride.
HAGEN M., STIRLING W.G., LANDER G.H., JONES D.L.

**4-04-182** **IN4, IN6**
Fe spin reorientation and RE spin dynamics.
LOEWENHAUPT M., SOSNOWSKA I.

**4-04-185** **IN12**
The magnetic phason mode in pure holmium.
PATTERSON C., MCMORROW D., BATES S., COWLEY R.A., PALMER S.B.

**4-05-140** **IN8**
Crystal field transitions in the intermediate-valence compound $YbCu_2Si_2$.
MITCHELL P.W., ROSS J.W., MURANI A.P., LLOYD R.G., CURRAT R.

**4-05-167** **IN4**
Inelastic neutron scattering in the Kondo lattice compound $CePt_2Si_2$.
GIGNOUX D., SCHMITT D., ZERGUINE M.

**4-05-177** **IN11**
Anomalous spin dynamics in the paramagnetic phase of spin glasses.
MEZEI F., MALETTA H., FARAGO B.

**4-05-194** **IN8**
Magnetic correlations and fluctuations under field in $CeRu_2Si_2$.
REGNAULT L.P., JACOUD J.L, ROSSAT-MIGNOD J., FLOUQUET J., LEJAY J.

**4-05-203** **IN4**
Spin dynamics of intermediate valent Eu-system.
HOLLAND-MORITZ E.

**4-05-214** **IN1**
High energy magnetic fluctuations in $La_2CuO_4$.
HAYDEN S. M., AEPPLI G., BROHOLM C., KJEMS J. K., REMEIKA M.

**4-05-218** **IN4**
Spin dynamics of Cu-moments in the high temperature superconductor $YBa_2Cu_3O_x$.
HOLLAND-MORITZ E., BLUMENROEDER S., ZIRNGIEBL E.

**4-05-222** **IN4**
Spin dynamics of $ReBa_2Cu_30_x$.
HOLLAND-MORITZ E., WALTER U., ERLE A., ZIRNGIEBL E.

**4-05-223** **IN13, IN10**
Oxygen diffusion in the high $T_c$ superconductor $YBa_2Cu_3O_{7-\delta}$.
PRANDL W., JANTSCH S., KEMMLER-SACK S.

**4-05-234** **IN11**
Search for spin-glass phase in $La_{2-x}Sr_xCuO_4$.
MALETTA H., MEZEI F., LARTIGUE C., FARAGO B.

# College 5

## Crystal and Magnetic Structures

### 5a-Crystallography

**5-11-165** **D19**
Crystallographic study of an (AlLiCuZn) single crystal of very large unit cell (a=13.9Å, c=82Å).
SAINFORT P., AUDIER M., DUBOST B., LANG J.M., CHEVRIER J., PANNETIER J.

**5-12- 93** **D19**
Structure of a non-centrosymmetric diacetylene.
BERTAULT M., HEYMAN M., SCHOTT M., STROHRIEGEL P., TOUPET L., CHEVRIER J.

**5-13-168** **D10, D19**
Location of hydride ligands in high coordination transition metal hydrides.
HOWARD J.A.K., SPENCER N., JOHNSON O., CRENNELL S.

**5-13-170** **D19**
Hydrogen bonding in α–cyclodextrin.$6H_2O$ clathrate.
LEBAS G., RYSANEK N.

**5-13-174** **D9**
Hydrogen bonding in $Ca(C_6(C_2H_5)_2O_4).3H_2O$.
ROBL C.

**5-13-175** **D19**
Beta-cyclodextrin ethanol complex.
SAENGER W., STEINER T., MASON S.A.

**5-13-178** **D9**
Neutron diffraction of $Ba(OH)_2.3H_20$ and $Ba(OH)_2.H_20$.
LUTZ H.D., ENGELEN B.

**5-13-180** **D9**
Single crystal structure determination of waterfree borate sodalite.
FELSCHE J., SCHEFER J.

**5-14-165** **D9**
X-N deformation density of 18-crown-6 with cyanamide, $C_{12}H_{24}O_6.2(H_2NCN)$.
LUGER P., BUSCHMANN J., DENNER L., KNOECHEL A.

**5-15-305** **S21**
Incommensurate phase of quartz.
BASTIE P., MOGEON F.

**5-15-307** **D15**
The P-T dependence of the structure of $PbHPO_4$.
PILTZ R.O., NELMES R.J, MCMAHON M.I.

**5-15-312** **D10**
P-T phase diagram of neutron incommensurate γ-phase of PAMC.
MOUDDEN A.H., VETTIER C.

**5-15-319** **D9**
High resolution study of the structure of squaric acid under pressure.
NELMES R.J., MCMAHON M.I., KUHS W.F.

**5-15-320** **D15**
The crystal structures of $D_2C_4O_4$ and $PbDPO_4$ at high pressure.
PILTZ R.O., MCMAHON M.I, NELMES R.J.

**5-15-321** **D10**
Examination of the charge-density wave in α-uranium.
MARMEGGI J.C., LANDER G.H., DELAPALME A.

**5-15-324** **D9**
Crystal structure of the high temperature superconductor $YBa_2Cu_3O_7$.
MCINTYRE G., RENAULT A., COLLIN G., POUGET J.P.

**5-15-333** **D9**
High resolution studies of (D)KDP.
NELMES R.J., MCMAHON M.I., HOWARD C.J., KUHS W.F.

**5-16-174** **D10**
Diffuse scattering and line broadening in $NaCN_{1-x}KCN_x$.
SCHRAEDER T., LOIDL A.

**5-16-176** **D9**
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HASTINGS J.B., SIDDONS D.P., LEHMANN M.S.

**5-16-179** **S21**
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BASTIE P., BELLET D.

**5-16-183** **D11**
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KRAETZIG E., MATULL R., RUPP R.A.

**5-16-184** **S21**
Asymptotic neutron Bragg scattering: neutron truncation rods from single crystal surfaces.
AL USTA K., DOSCH H., PEISL J.

**5-16- X** **S18**
Test of phase shifter materials for future Fizeau experiments.
BONSE U., BRINKMANN J.

**5-16-Y** **S18**
Test of the double crystal interferometer set-up.
BONSE U., UEBBING H.

**5-21-270** **D2B**
Direct determination of zeolite crystal structures from neutron powder diffraction data.
ADAMS J.M., GOLAB M.

**5-21-338** **D1B, D2B**
The low-temperature phases of $SF_6$.
COCKCROFT J. K., FITCH A. N.

**5-21-346** **D1B**
Hydrogen absorption characteristics of hyperstoichiometric laves phase compounds.
FRUCHART D., ROUAULT A., PONTONNIER L., TRIANTAFILLIDIS G., FRUCHART R.

**5-21-348** **D1A**
Refinement of the crystal structure of the new perovskite $Sr(Na_{1/4}Sb_{3/4})O_3$.
ALONSO A.J., MZAYEK E., RASINES I.

**5-21-353** **D2B**
Structure determination of some ternary oxide phases.
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***5-21-355** **D1B, D1A**
Structure of $H_2Ti_3O_7$ ramsdellite and study of its thermal decomposition.
LE BAIL A., FOURQUET J.L.

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Neutron diffraction study of the fast ionic conductors $Li_2ZnCl_4$ and $Li_2MnBr_4$.
KUSKE P., KELLERSOHN T.

**5-21-360** **D2B**
Neutron powder diffraction studies of zeolite ZK-5.
FISCHER R.X., TILLMANNS E., SHANNON R.D., HEWAT A.

**5-21-372** **D2B UNP**
Structure determination of $Bi_3BiO_7$.
JANSEN M., BEGEMANN B.

**5-21-373** **D2B**
Determination of interstitial site occupation in $Zr_2NiH_x$ hydride.
CHIKDENE A., BAUDRY A., BOYER P., MIRAGLIA S., FRUCHART D., SOUBEYROUX J.L.

**5-21-376** **D2B**
High-resolution neutron diffraction study of $TlAu(CN)_2$.
FISCHER P., LUDI A., PATTERSON H.

**5-21-378** **D2B**
The crystal structure of acetylacetone.
HORSEWILL A.J., KEMP A., AIBOUT A.

**5-21-381** **D2B**
Powder studies on aluminate sodalite.
DEPMEIER W., MELZER R.

**5-21-382** **D2B**
Lithium aluminosilicates.
WELLER M.T., WONG G.

**5-21-383** **D2B**
Neutron diffraction studies of zeolite ZK-5, mordenite, and erionite.
FISCHER R.X., TILLMANNS E., SHANNON R.D., HEWAT A.

**5-21-384** **D2B**
Crystal structure of the catalytic important zeolites of ferrierite, offretite and erionite type.
FJELLVÅG H., KJEKSHUS A., NORBY P.

**5-21-386** **D1A**
Powder diffraction of p-xylene and benzene absorbed on faujasite-type zeolite.
FUESS H., CZJZEK M.

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**5-21-394** **D1B**
Structural phase transition in $NaCN_{1-x}KCN_x$.
SCHRAEDER T., LOIDL A.

***5-21-397** **D2B**
Phase transitions in leucite.
BOYSEN H., PENTINGHAUS H.

**5-21-404** **D2B**
Powder measurement of super- and non-superconducting oxides $La_{2-x}Sr_xCu_{1-y}Co_y0_4$.
HOHLWEIN D., HOSER A., SONNTAG R., PRANDL W., KEMMLER-SACK S.

**5-21-408** **D1A, D1B**
Structural phase transitions in deuterated cyclohexane, $C_6D_{12}$.
CRAVEN J., HATTON P.D., HOWARD C.J., PAWLEY G.S., CRIADO A.

**5-21-410** **D2B**
Structure determination of two allotropes of polymeric sulfur $S\omega_1$ and $S\omega_2$.
JUMAS J.C., OLIVIER-FOURCADE J., MICHAUD F.

**5-21-413** **D2B**
Phase transitions in NaOD and in NaOH.
HOWARD C.J., NELMES R.J., BASTOW T.J.

**5-21-414** **D2B**
Phase transitions in RbOH and RbOD.
HOWARD C. J., HATTON P.D., BASTOW T. J., NELMES R. J.

**5-21-431** **D2B**
Structural analysis of cesium substituted barium hollandites.
CHEARY R.W.

**5-21-432** **D1A**
High-resolution neutron-diffraction study of the ionic conductor $\beta$-$LiNaSO_4$.
FISCHER P., GRANELI B., BRINKMANN D.

**5-21-438** **D1A**
Solid-state modification of zeolites: second generation zeolitic catalysts.
GLADDEN L.F., BARRIE P.J., KLINOWSKI J.

**5-21-447** **D2B**
Structure/thermal treatment relations in the "123" high temperature superconductor.
GREAVES C., SLATER P.R.

**5-21-TEST** **D1A, D2B**
Debye waller factors in heavy fermion superconductors.
NEUMANN K.U., ZIEBECK K.R.A., CAPELLMANN H.

**5-22-283** **D1B**
Crystallographic phase transitions in the system $(Fe_{1-x}Mn_x)_2P$ $(0 < x < 1)$ at high temperature.
CHENEVIER B., BACMANN M., FRUCHART D., SOUBEYROUX J.L.

**5-22-317** **D1A**
Structural mapping of the $H_20$ and $D_20$ ice phase diagrams.
FINNEY J.L., KUHS W.F., LONDONO D., BOUQUIERE J. P.

**5-22-325** **D1B**
Phase transition study in $LiMnFeF_6$.
COURBION G., FEREY G.

**5-22-332** **D1B**
In situ reduction of $Ba_2YCu_30_7$ superconductor : thermodiffractometry study.
RODRIGUEZ J., BASSAS J., OBRADORS X., VALLET M., CALBET J., ANNE M., PANNETIER J.

**5-22-333** **D1B**
A sol-gel route to high $T_c$ superconductors.
SIMON C., ROSENMAN I., VALENTE I., CONVERT P.

**5-22-338** **D1B**
Etude cinétique de la réaction à haute température entre AlN et $TiO_2$.
MOCELLIN A., MANGIN P., CASTANO J., MOECKLI P., SPERISEN T.

**5-22-339** **D1B**
A dynamic neutron diffraction study of hydrothermal syntheses of alumino-silicate structures using a special cell.
BARNES P., TARLING S. E., POLAK E., MUNN J., CLARK S.M.

**5-22-341** **D1B**
Time resolved neutron diffraction studies of crystalline phases in hydrating OPC Cement.
ALLEN A. J., WILDING C. R., GROVES G.

**5-22-348** **D1A**
An investigation of defect structures in fluorite related nitride fluorides of Ce and Pr.
SCHWEDA E.

**5-23-300** **D20**
Short range order in Ti-Fe alloys.
COWLAM N., GYORFFY B.L., ZIEBECK K.R.A., IVISON P.K., CONVERT P.

**5-23-305** **D11**
Observation of platelets in diamond.
DAVIES G., WILKINSON C., EVANS T.

**5-23-312** **D20**
Study of 5000 year-old bones.
BACON G.E.

**5-23-313** **D1A**
Residual stress distribution in a ceramic bending bar.
PINTSCHOVIUS L., FETT T., MUNZ D.

**5-23-315** **D1A**
Residual stress in generic engineering components.
(a) Railway rail slices.
WEBSTER P.J., WEBSTER G. A., LOW K. S, MILLS G., BOURKE M. A. M., McGILLIVRAY H. J.

**5-23-317** **D1A**
Stress field in weldments and around cracks.
HUTCHINGS M.T., ALLEN A J., DAWES S.J.

**5-23-318** **D1B**
Neutron texture analysis of deformed granitic rocks.
WENK H.R.

**5-23-321** **D1B**
Preferred orientation of l herzolithe.
BROKMEIER H.G., BUNGE H.J., RITTER C.

**5-23-325** **D1A**
Residual stress in generic engineering components.
(b) An electron beam weld.
WEBSTER P.J., LOW K.S., MILLS G., WEBSTER G.A., BOURKE M.A.M. McGILLIVRAY H.J.

**5-23-327** **D1A**
Anisotropic lattice strains in compressionally stressed polycrystalline samples.
HUTCHINGS M.T., WINDSOR C.G., DAWES S.J.

**5-25-190** **D20**
Texture and age in the bones of sheep.
BACON G.E.

## 5b-Magnetism

**5-31-240** **D20**
Magnetic field dependence of the antiferromagnetic structure of neodymium.
FORGAN E.M., GIBBONS E.P., MCEWEN K.A., LEE S.L.

**5-31-244** **D1B**
$Cu_{0.5}In_{0.5}Cr_2Se_4$, a spin glass with helical ordering?
PLUMIER R., SOUGI M.

**5-31-248, 5-32-321** **D10**
Magnetic transition in $Ce_2Sn_5$ and $Ce_3Sn_7$ under applied field.
STUNAULT A., RESSOUCHE E., GIVORD F.

**5-31-275** **D2B**
$Mn_2As_2O_7$.
BUCHLEY A., BRAMWELL S., DAY P.

**5-31-286** **D20, D2B**
Magnetic ordering in $REP_5$ (RE=Ce, Nd).
CHATTOPADHYAY T., HARTWEG M., VON SCHNERING H.G.

**5-31-295** **D2B**
Para-ferro-antiferromagnetic transition in a disordered system; $Ce(Fe_{1-x}Co_x)_2$.
COLES B.R., KENNEDY S.J., ROY S.B.

**5-31-313** **D2B**
Spin glass antiferromagnetic ordering in $FeSbO_4$.
OBRADORS X., RODRIGUEZ J., PANNETIER J., BASSAS J.

**5-31-314** **D2B**
Cationic distribution delta spin reorientation in $BaFe_{12-2x}Co_xM_x0$ (M=Te, Sn, Mn) hexagonal ferrites.
OBRADORS X., RODRIGUEZ J., PERNET M., VALLET M., COLLOMB A., BATLLE X.

**5-31-320** **D2B**
A neutron powder diffraction study of the magnetic order of the antiferromagnet $PrCo_2P_2$.
REEHUIS M., MOELLER M.H., JEITSCHKO W.

**5-31-324** **D2B**
Superconductivity and magnetic order in $Pd_2RSn$ intermetallics.
CRANGLE J., WEBSTER P., ZIEBECK K.

**5-31-339** **D17**
Microcrystal size of the Suzuki phase.
RODRIGUEZ F., MORENO M, GOMEZ SAL J.C., JANOT C.

**5-31-341** **D1B, D2B**
Determination of the magnetic structures of the layer phosphates $M^IM^{III}PO_4.D_2O$.
CARLING S.G., VISSER D., DAY P.

**5-31-345** **D1B**
Thermodiffractometric studies on some deuterated Mn(III) aquatetrafluorides.
PALACIO F., RODRIGUEZ J., PANNETIER J.

**5-31-346** **D2B**
High-resolution neutron-diffraction study of para- and 3D antiferromagnetic $Tb_2Cl_3$.
SCHMID B., FISCHER P., KREMER R., SIMON A., MATTAUSCH J.

**5-31-351** **D2B**
Hydrogen localisation and its influence on the magnetic structure and anisotropy of $Ho_2Fe_{14}B_{11}H(D)_x$.
RILLO C., FRUCHART D., MIRAGLIA S., WOLFERS P.

**5-31-353** **D1B**
Origin of pressure effect on magnetisation in UIr and UPt.
FRINGS P., DOMMANN A.

**5-31-354** **D1B**
Determination of the magnetic structure of β-phase cerium and cerium-neodymium alloys.
GIBBONS E.P., FORGAN E.M., LEE S., McEWEN K.A.

**5-31-355** **D1B**
Magnetic structures of $Ce_{1-x}Pr_xAl_2$.
MARSHALL W.G., McEWEN K.A.

**5-31-357** **D1B**
Magnetic structures of rare earth disilicides.
PIERRE J.

**5-31-362** **D4**
Neutron diffraction study of the magnetic behaviour of $Gd_2In$.
BOUREE F., ACHARD J.C., RAVOT D.

**5-31-367** **D17**
Finite size effects in magnetic graphite intercalated compounds.
ROGERIE J., SIMON C.

**5-31-368** **D17**
Magnetic correlations in random anisotropy $Dy_xY_{1-x}Al_2$ magnets.
DEL MORAL A., SCHWEIZER J., ARNAUDAS J.I., IBARRA M.R., ALGARABEL P.A.

**5-31-369** **D17**
Magnetic structures in the phase diagram of $GdAg_{1-x}Zn_x$.
KOEBLER U., ZINN W.

**5-31-372** **D1B**
Antiferromagnetism and oxygen deficiency in $La_2NiO_{4-y}$.
SAEZ PUCHE R., MARTINEZ J.L., RODRIGUEZ-CARVAJAL J.

**5-31-374** **D2B**
Temperature dependence of spin density wave in $La_2CuO_4$ and non-superconducting $La_{1.97}Sr_{0.03}CuO_4$.
ROSSEINSKY M.J., PRASSIDES K., DAY P.

**5-31-375** **D15**
Magnetic ordering of the high temperature superconductor $GdBa_2Cu_30_{6.5}$.
CHATTOPADHYAY T., BROWN P.J., MALETTA H.

**5-31-377** **D1**
Lattice structure of vortex lines in $Ba_2HoCu_30_7$ superconductor.
RODRIGUEZ J., OBRADORS X., GARCIA J.L., JANOT C., TASSET F., LABARTA A., VALLET M., CALVET J.G.

**5-31-382** **D4B**
Magnetic structures of the layered rare-earth halide compounds: $RXD_y$ and $RE_2XC$ (X=C1, Br, I, RE=Gd).
SIMON A., BAUHOFER W., MATTAUSCH H.J., ARONS R.R., COCKCROFT J.K.

**5-31-384** **D1B**
Investigation of magnetic ordering in Nd-La alloys by powder diffraction.
FORGAN E.M., LEE S.L., MARSHALL W.G , McEWEN K.A.

**5-31-386** **D1B**
Magnetic crossover in $Ce_{1-x}Pr_xAl_2$, $0.08 \leq x \leq 0.15 \leq$
MARSHALL W.G., MCEWEN K.A.

**5-31-391** **D20**
Magnetic order of some compounds in the ZrNiAl structure.
MALETTA H., DE BOER F.R., SECHOVSKY V.

**5-31-397** **D1B**
Magnetic order in superconducting Heusler alloys of type $Pd_2(R_1R_2)Sn$.
CRANGLE J., WEBSTER P., ZIEBECK K.

**5-31-406** **D2B**
Cationic distribution and spin reorientation in $BaFe_{12-2x}Co_xTi_x0_{19}$ hexagonal ferrites.
OBRADORS X., BATLLE X., RODRIGUEZ J., GARCIA J.

**5-31-408** **D1B**
Competing magnetic order in the garnets calderit and $Cr_2(CaNaEr)Ge_30_{12}$.
BRUECKEL T., HOCK R., FUESS H., PRANDL W.

**5-31-412** **D17**
Magnetic clusters in $FeAl_{1-x}Cu_x$ alloys.
BOOTH J.G., LAWSON P., DE BOER F.R., FRINGS P.

**5-31-414** **D17**
Magnetic instability of Pd films.
BLAND J.A.C., JOHNSON A.D., NORRIS C., LAUTER H.J.

**5-31-415** **D17**
Ferromagnetic to antiferromagetic transition in epitaxial Cr films.
BLAND J.A.C., JOHNSON A.D., NORRIS C., LAUTER H.J.

**5-32-249, 5-32-300** **D10**
Magnetic order in CeNd alloys.
MCEWEN K.A., FORGAN E.M, GIBBONS E.

**5-32-290** **D15**
Crystallographic structure of the high pressure phase of a single crystal of CeNi.
GIGNOUX D., VOIRON J.

**5-32-293** **D9**
Magnetic structure of $Ho_2Fe_{14}B$ study of the reorientation phenomena.
WOLFERS P., FRUCHART D., BARTOLOME J., L'HERITIER P., YAOUANC A.

**5-32-297** **D15**
Commensurate-incommensurate coexistence in Cr-O.6 at.%Si.
PALMER S.B., MELVILLE R.J.

**5-32-300** **see** **5-32-249**

**5-32-304** **D15**
Antiferromagnetism in copper phosphate $Cu_3(PO_4)_2$.
FORSYTH J.B., WILKINSON C., PASTER S.

**5-32-308** **D15**
Magnetic phase diagram of $FeI_2$ at very low temperatures.
WIEDENMANN A., REGNAULT L.P.

**5-32-312** **D10**
Diffuse critical scattering in $MnS_2$.
CHATTOPADHYAY T., BURLET P., BRUECKEL T.

**5-32-314** **D15**
Magnetic structure of $MnS_2$: single-q or triple-q.
CHATTOPADHYAY T., BURLET P.

**5-32-315** **D15**
Effect of hydrostatic pressure on the incommensurate phase of $EuAs_3$.
CHATTOPADHYAY T., BROWN P.J.

**5-32-317** **D16**
Magnetic structure of a single crystal line $CoCl_2$-$GaCl_3$ ($CoCl_2$-$AlCl_3$) Bi-intercalated graphite.
ROGERIE J., SIMON C.

**5-32-319** **D15**
Examination of critical scattering in NpSb in the single and triple k states.
PLESKA E., BURLET P., LANDER G.

**5-32-320** **D10**
Commensurate-incommensurate coexistence in Cr-0.6 at %Si.
PALMER S.B., MELVILLE R.J., SOUSA J. B.

**5-32-321** **see** **5-31-248**

**5-32-322** **D16**
Search for long range order in Sc-Er alloy.
CAUDRON R., PAPPA C.

**5-32-324** **D10**
The quadruple-q magnetic structure of neodymium.
GIBBONS E.P., FORGAN E.M., LEE S.L., McEWEN K.A.

**5-32-325** **D10**
Investigation of magnetic field effects on magnetic ordering of neodymium, using an in situ applied field in an orange cryostat.
FORGAN E.M., LEE S.L., GIBBONS E.P., McEWEN K.A.

**5-32-338** **D15**
Magnetic order in holmium trifluoride, $HoF_3$, a singlet ground state system.
LEASK M.J.M., WELLS M..R.

**5-32-340** **D9**
Hot neutron diffraction study of single crystal $Gd_2Cl_3$.
KREMER R.K., MATTAUSCH H.J., SIMON A., COCKCROFT J.K.

**5-32-341** **D10**
Elastic diffuse magnetic scattering from an antiferromagnet with degenerate ground state.
BRUECKEL T., PRANDL W.

**5-32-243** **D15**
Examination of the structure as function of temperature of single crystal of $La_2NiO_4$.
LANDER G.H., BROWN P.J., HONIG J.M.

**5-32-348** **D15**
Magnetic ordering of the high temperature superconductor $ErBa_2Cu_30_{7-\delta}$.
CHATTOPADHYAY T., BROWN P.J., MALETTA H.

**5-32-349** **D9**
Crystal and magnetic structure $GdBa_2Cu_30_{7-\delta}$.
CHATTOPADHYAY T., MALETTA H., BROWN P J.

**5-32-350** **D20**
Magnetic ordering in the high temperature superconductor $ErBa_2Cu_30_{7-\delta}$.
MALETTA H., CHATTOPADHYAY T., BROWN P.J.

**5-32-352** **D11**
Search for SANS due to flux lines in the mixed state of high-$T_c$ superconductors.
FORGAN E.M., PAUL D. MCK, MITCHELL P., MOOK H.

**5-33-202** **D3**
Diamagnetism in naphthalene.
WILKINSON C., FORSYTH J.B., BROWN P.J.

**5-33-218** **IN20**
Dynamic form factor of Invar alloys.
ZIEBECK K.R.A., MANKIKAR M., JASSIM I.K., BROWN P.J.

**5-33-224** **D15, D3**
Spin density in copper nitroxid complexes.
SCHWEIZER J., REY P., RESSOUCHE E., BOUCHERLE J.X., GRAND A.

**5-33-227** **D3**
Structure and magnetisation density of ferrous ammonium tutton salt at low temperature.
FORSYTH J. B., FIGGIS B. N., KUCHARSKI E. S.

**5-33-227** **D15**
Structure and magnetisation density of ferrous ammonium tutton salt at low temperature.
FORSYTH J.B., FIGGIS B.N., KUCHARSKI E.S.

**5-33-235** **D3**
Form factor in the anisotropic compound $Ce_3Sn_7$.
GIVORD F., BOUCHERLE J.X., STUNAULT A., SCHWEIZER J., LEJAY P.

**5-33-236** **D9, D3**
Use of Schwinger scattering to probe acentric electron distributions in III-V compounds.
BROWN P.J., FORSYTH J.B.

**5-33-237** **IN20**
Determination of the magnetic profile of multilayers Fe/Si.
MANGIN P., BRUSON A., MARCHAL G., DUFOUR C., ERWIN R., RHYNE J.J., VETTIER C.

**5-33-240** **D9, D3**
Schwinger scattering to probe acentric electron distribution in III-V compounds.
FORSYTH J.B., BROWN P.J.

**5-33-248** **D3**
Examination of the spin density in single crystals of $La_2NiO_4$.
LANDER G.H., HONIG J.M.

**5-33-254** **D3B**
Magnetic form factor of copper in $YBa_2Cu_3O_{7-\delta}$
BOUCHERLE J.X., HENRY J.Y., JURGENS M., ROSSAT-MIGNOD J., SCHWEIZER J.

**5-33-TEST** **D3B**
Applied field study of $Gd_{64}Sc_{36}$.
MELVILLE R.J., ECCLESTON R.S., PALMER S.B., VRTIS M.L.

**5-34-X** **IN3, IN8, IN20, D10**
Magnetic phase diagram of $YBa_2Cu_3O_{6+x}$.
ROSSAT-MIGNOD J., BURLET P., JURGENS M.J., REGNAULT L.P., VETTIER C.

**5-34-239** **D7**
Paramagnetic scattering from ZrZn2.
WILLIAMS D.E.G., CRANGLE J.

**5-34-250** **IN20**
The absolute magnetic configuration of $LiCoPO_4$ and its temperature dependence.
FORSYTH J.B., BROWN P.J., TASSET F.

**5-34-251** **IN20**
Ferromagnetic moment and magnetic structure of single-crystal Co/Cu superlattices.
MIRANDA R., BATALLAN F., CEBOLLADA A., MARTINEZ J.L.

**5-34-253** **IN20**
Paramagnetic scattering in weakly ferromagnetic $Y(Co_{1-x}Al_x)_2$ alloys.
COLES B.R., KENNEDY S.J.

**5-34-261** **D7**
Energy spectrum of the magnetic fluctuation in $YBa_2Cu_3O_6$.
BRUECKEL T., SCHAERPF O., CAPELLMANN H., KEMMLER-SACK S., COMBERG A., PASSING H.

**5-34-262** **D7**
Polarized neutron scattering at Er-Ba-Cu and Ho-Ba-Cu high $T_c$ superconductors.
ZEILINGER A., WEBER H.W., SCHAERPF O., HUYN O.B., FINNEMORE D K.

**5-35- 71** **S20**
Quantitative neutron topography and local diffraction measurements using a good resolution PSD.
BARUCHEL J., KURODA K., MICHALOWICZ A., SILLOU D., SCHLENKER M.

**5-41- 8** **S20**
Study of the dendritic texture of nickel based single crystal superalloy by means of neutron topographies.
BASTIE P., BELLET D.

# College 6

## Liquids, Disordered Materials and Metal Physics.

**6-00- 2** **D11**
Carbon clusters in kapton.
FINK D., IBEL K.

**6-01-105** **IN6**
Ripplons in $^4$He-films.
FRANK V., GODFRIN H., LAUTER H.J., LEIDERER P.

**6-01-107** **IN6**
Pressure dependence of S(Q, ω) in $^3$He-$^4$He mixtures.
SCHERM R., GUCKELSBERGER K., KOERFER M., HENNECKE F., HEINEMANN W., FAK B., DIANOUX A.J., STIRLING W., BLANC Y., RAGAZZONI J.L.

**6-01-112** **IN12**
Temperature dependence of S(Q, ω) for liquid $^4$He.
II Small wavevectors, Q.
STIRLING W.G., GLYDE H.R.

**6-01-TEST** **IN5**
Report on test experiment on neutron Brillouin scattering.
EGELSTAFF P.A., YOUDEN J., SUCK J.B., KEARLEY G.

**6-02- 74** **D4B**
Structure of gaseous argon at low densities and extraction of the pair potential.
FREDRIKZE H., DE GRAAF L.A., VAN WELL A.A., VAN TRICHT J.B., BAROCCHI F., MAGLI R., CHIEUX P.

**6-02- 76** **IN12**
Two-dimensional density fluctuations in graphite intercalates.
WHITE J.W., HIRST P.R., MAGERL A., HAYDEN S.

**6-02- 78** **D 7**
Polarization analysis of inelastic neutron scattering on liquid lithium.
MORKEL C., SEDLMEIR J.

**6-02-TEST** **IN15**
Neutron inelastic scattering at very small momentum transfers.
EGELSTAFF P.A., SUCK J.B.

**6-03-154** **IN5**
Dynamics of molten AuCs alloys.
McGREEVY R., VAN DER LUGT W.

**6-03-157** **D4B**
Resonant states and local ordering in liquid alloy $Al_{80}Ni_{20}$ alloy.
MARET M., PASTUREL A., POMME T., SENILLOU C.

**6-03-160** **D4B**
The structure of liquid and glassy $GeSe_2$ by isotopic substitution.
SALMON P.S., PENFOLD I.T.

**6-03-161** **D4B**
The structure of liquid Cu-Se alloys.
BARNES A.C., ENDERBY J.E.

**6-04- 31** **D4B**
Structure factor of DCl over a broad range of density up to supercritical temperatures.
BERTAGNOLLI H., TOEDHEIDE K., HOFFMANN M., HUMMEL I.

**6-04- 36** **D4**
Hydrophobic hydration by nitrogen isotope substitution: concentration and ion-pairing effects.
FINNEY J.L., TURNER J.

**6-04- 45** **D4B**
Structural studies of liquid sulphur dioxide and its ammonia solutions.
BERMEJO F.J., ENCISO E., ALVAREZ M., GARCIA N.

***6-04- 53** **D4B**
The liquid structure of haloethane by neutron diffraction.
BERMEJO F.J., ENCISO E.

**6-05-113** **D20**
Polar group hydration using nitrogen isotope substitution: urea in $H_2O/D_2O$ mixtures.
FINNEY J.L., TURNER J., NEILSON G.W.

**6-05-119** **D20**
Neutron diffraction study of the eutectic melt of ammonium nitrate with calcium nitrate.
ADYA A.K., NEILSON W.

**6-05-120** **IN6**
The dynamics of water molecules in aqueous ionic solution: the rotational motion.
SALMON P.S.

**6-05-129** **IN6**
Inelastic neutron scattering from molten nickel iodide.
HOWE R.A., MCGREEVY R.L., WOOD N.D.

**6-05-135** **IN10**
The dynamical properties of the $Cr^{3+}$ ion in aqueous solution: and incoherent quasi-elastic neutron study.
SANDSTROM M., NEILSON G.W., SALMON P.S., HERDMAN G.J.

**6-05-137** **D4**
Hydrophobic hydration by nitrogen isotope substitution: tetramethyl ammonium chloride in $H_2O/D_2O$ mixture.
FINNEY J.L., TURNER J.

**6-05-142** **IN6**
Collective modes in molten LiCl.
MCGREEVY R., PUSZTAI L.

**6-05-143** **IN8, IN6**
Dynamic structure factor of liquid metal-molten salt mixtures.
JAL J.F., MATHIEU C., DUPUY J., SUCK J.B., CHIEUX P.

**6-05-144** **IN10**
The dynamics of $Cu^{2+}$ and $Ni^{2+}$ methanol solutions.
SALMON P.S., LOND P.B.

**6-05-146** **IN6**
Time resolved study of free and bound water during cement hydration.
ALLEN A.J., WILDING C.R., GROVES G., RODGER S.A.

**6-05-147** **D11**
Growth process and structure of gels.
DAUGER A., SMAIHI M., BOILOT J.P.

**6-05-149** **D4B**
Neutron diffraction study of molten ammonium nitrate.
ADYA A.K., NEILSON G.W.

**6-05-157** **D4B**
The hydration of samarium(III) in aqueous solution.
MERBACH A.E., COSSY C., ENDERBY J.E., BARNES A.C.

**6-05-164** **D20**
Aqueous solutions at high pressure and temperature.
NEILSON G.W., HOWE M.A., BROADBENT R.D., FRANCK E.U.

**6-05-167** **D4B**
The hydration of lanthanide (III) ions in aqueous solution.
MERBACH A.E., COSSY C., ENDERBY J.E., BARNES A.C.

**6-06- 73** **IN11**
Investigation of the dynamics of liquid-glass transition by NSE and TOF spectroscopy.
MEZEI F., KNAAK W., FARAGO B.

**6-06- 79** **D11**
Permeation of $D_2O$ through hydrated cement systems.
ALLEN A.J., WILDING C.R., GROVES G., RICHARDSON I.

**6-06- 89** **D1B**
Amorphisation reaction of $Zr_3Rh$-D alloys.
COWLAN N., TITMAN J.M., WRIGHT M.S.

**6-06- 95** **IN10**
Local and long-range motions in $Ag^+$ and $Cu^+$ superionic glasses.
TACHEZ M., MERCIER R., MALUGANI J.P., ROUSSELOT C.

**6-06- 96** **D4B**
Neutron diffraction with amorphous V-Zr and Ni-Zr-alloys.
RUTH W., KUSCHKE M., LAMPARTER P., STEEB S.

**6-06-112** **D11**
Glass transition study.
DUPUY J., JAL J.F., CARMONA P., AOUIZERAT A., CHIEUX P.

# Experiments Carried Out at the ILL

**6-06-122** **IN11**
Study of the glass transition in polybutadiene.
FRICK B., RICHTER D.

**6-06-123** **IN13**
Transition from localized sub-$T_g$ motion (β-process) to diffusive motion (α-process) in a molecular glass.
FUJARA F., KIEBEL M., PETRY W.

**6-06-126** **IN13**
Dynamic critical exponents of the glass transition.
JUNG S.

**6-06-127** **IN6**
Inelastic neutron scattering study of medium-range order and low-energy excitations in amorphous $Ge_xSe_{1-x}$ alloys.
ELLIOT S.R., GLADDEN L.F., WRIGHT A.C., SINCLAIR R.N.

**6-06-129** **IN6**
The vibrational spectrum of amorphous semiconducting alloys.
ROSENBERG H.M., NEEDHAM L.M.

**6-06-130** **IN4**
Vibrational properties of $Se_{1-x}Ge_x$ and $Te_{1-x}Si_x$ glasses.
GOMPF F., KAMITAKAHARA W.A., CAPPELLETTI R.L., GOMPF F.

**6-06-139** **D4B**
Short and medium range order in $Al_6CuLi_3$ quasicrystals.
DUBOIS J.M., BOISSIEU M. DE, AUDIER M.

**6-06-141** **D4B**
Structural relaxation in $Ge_xSe_{1-x}$ chalcogenide glasses.
MORA M.T., TORRELLES X., CLAVAGUERA N.

**6-06-142** **D4**
Local environment of $Li^+$ and $Ca^{++}$ in silicate glasses.
GASKELL P.H., ECKERSLEY M.C.

**6-06-143** **D17**
SANS with $(FeNi)_xB_{100-x}$ metallic glasses.
TRAEUBLE H., LAMPARTER P., STEEB S.

**6-06-146** **D2B, D20, D1B**
Structural relationships between quasiperiodic and amorphous AlMnSi and AlCrSi and related phases.
AUDIER M., BOISSIEU M. DE, DUBOIS J.M., JANOT C.

**6-06-149** **D11**
Short and medium range magnetic order in the amorphous alloys $Tb_xCu_{1-x}$ (x =. 5;. 65).
BOUCHER B., SANQUER M., TOURBOT R., CHIEUX P.

**6-06-150** **IN11**
Investigation of the dynamics of the self-correlations near the glass transition.
MEZEI F., KNAAK W.

**6-06-154** **IN13**
Phonon fracton crossover in silica aerogels.
BUCHENAU U., CONRAD H., FRICK B., RICHTER D., FRICKE J., REICHENAUER G.

**6-06-156** **IN6**
The structure and dynamics of amorphous Si-D, Si-H and Si-F II
WRIGHT A.C., SINCLAIR R.N., JANSEN F., HANNON A.C.

**6-06-162** **D4**
The structure of amorphous diamond films.
GASKELL P.H., SAEED A., MCKENZIE D.R.

**6-07-100** **D2B, D10**
Comparative crystallographic study of an $Al_6CuLi3$ single quasicrystal and of a "cousin" $Al_5CuLi_3$ single crystal of cubic structure by neutron diffraction.
SAINFORT P., DUBOST B., AUDIER M., BOUVAIST J., JANOT C.

**6-07-122** **D11**
Irradiation influence on the decomposition of alloys.
WAGNER W., WIEDENMANN A.

**6-07-123** **D7**
Diffuse scattering of Co in β-Zr.
HEIMING A., PETRY W., TRAMPENAU J., VOGL G.

**6-07-124** **D10**
Study of vacancy short-range-order and atomic displacements in $NbC_{0.73}$.
NOVION C.H. DE, PRIEM T., BEUNEU B., LEFEBVRE S., CHRISTENSEN A.

**6-07-127** **D10**
Diffuse scattering of Co in β-Zr.
HEIMING A., PETRY W., TRAMPENAU J., VOGL G.

**6-07-128** **D11**
Small angle scattering from Ge-PbBi alloys.
COWLAM N., STEWART R.J., MESSOLORAS S.

**6-07-129** **D11**
Coherent precipitates with variable lattice mismatch in Ni-Al-Mo single crystals.
KOSTORZ G., BAENNINGER R., CALDERON H., DUBEY P., PELLEGRINI J.

**6-07-131** **D1B**
Structure determination of the intergrain precipitations in the shape memory alloy NiTi.
STOEHR C., TIETZE H., GEICK R.

**6-07-132** **D11**
Thermally induced precipitations in the shape-memory alloy NiTi.
STOEHR C., TIETZE H., GEICK R.

**6-07-133** **IN8**
Search for change in soft ω mode and ω-phase particles in ZrCo.
HEIMING A, PETRY W., TRAMPENAU J., VOGL G.

**6-07-134** **IN13**
Origin of the strong Debye-Waller factor of nanocrystalline materials.
WALLNER G., PEISL J., HERR U., GLEITER H.

**6-07-136** **IN10**
Atomic motion in an icosahedral AlLiCu quasicrystal.
JANOT C., MAGERL A., DUBOIS J.M.

**6-07-138** **D1B**
Real time study of the growth of icosahedral crystals by solid state reaction.
DUBOIS J.M., BOISSIEU M. DE, DONG C., JANOT C.

**6-07-149** **D17**
Influence of plastic strain amplitude in the stability of spinodal microstructure of fatigued superferritic stainless steels.
ISALGUE A., ANGLADA M.

**6-07- X** **S30**
Depth profiling of light elements in solids.
FINK D., WANG L., OBERSCHACHTSIEK P., ROSNY M. DE.

**6-08-244** **D10**
Coherent quasielastic line width in $NbD_x$.
DOSCH H., BURKEL E., PEISL J.

**6-08-251** **IN10**
Search for high temperature superionic behaviour in metal hydrides.
BARNFATHER K.J., STYLES G.A.

**6-08-256** **IN 6**
Pressure dependence of hydrogen tunneling in niobium.
WIPF H., STEINBINDER D., WEISS G., NEUMAIER K., DIANOUX A.J., MAGERL A., VETTIER C.

**6-08-260** **IN5**
Tunneling of hydrogen in $Nb(CH)_x$.
NEUMAIER K., GUTSMIED P., WIPF H., STEINBINDER D., BLANK H., KEARLEY G.

**6-08-261** **IN6, IN5**
Cluster complexes with interstitial hydrogens: models for hydrogen in metals.
JAYASOORIYA U.A., JOHNSON B.F.G., DALLIN P.

**6-08-263** **IN10**
High-temperature anomalous H diffusion in $YH_x(ScH_x, ZrH_x; 1.8 \leq x \leq 1.9)$.
STEINBINDER D., WIPF H.

**6-08-264** **IN8**
Quasi-elastic investigation of time-dependent correlation in the diffusion of hydrogen in single crystal $\alpha'$-$NbH_{0.79}$ at 548K.
COOK J.C., RICHTER D., ROSS D.K., HEMPELMANN R.

**6-08-265** **IN1**
Hydrogen vibrations in hydrides of Mo, Cr, Ni and alloys of $V_{.9}Ru_{.1}$ and $Nb_{.75}Ru_{.25}$.
ANTONOV V.E., PONYATOVSKY E.G., BOKHENKOV E.L., BELASH I.T.

**6-08-271** **D4B**
The structure and dynamics of amorphous Si-D, Si-H and Si-F I.
WRIGHT A.C., SINCLAIR R.N., JANSEN F., CHIEUX P., BELLISSENT R.

**6-08-281** **D11**
Small angle neutron scattering study of the distribution of hydrogen in low carbon steel.
ROSS D.K., IORDANOVA I., WINDSOR C.G., FORCEY K.F., HOWE L.D., COOK J.C.

**6-09- 37** **IN6**
Oxygen diffusion in $Sr_2Co_2O_5$.
ANNE M., RODRIGUEZ J., PANNETIER J., OBRADORS X., GONZALES-CALBET J.

**6-09- 40** **D9**
Defect clustering in uranium dioxide.
WILLIS B.T.M., CATLOW C.R.A., MURRAY A.D.

**6-09- 43** **D20**
The structure of the fast ion conductors AgBr and AgCl.
KEEN D.

**6-09- 44** **IN6**
Dynamics of $Na^+$ in a single crystal of $Na\beta''Al_2O_3$.
FANJAT N., LUCAZEAU G., BATES J., DIANOUX A.J.

**6-09- 47** **D10**
Quasielastic linewidth of Huang scattering from reorienting defects $(KBr)_{1-x}(KCN)_x$.
WOCHNER P., BURKEL E., PEISL J.

# College 8

## Biology.

***8-01- 82** **DB21**
Crystallographic studies of a membrane protein: the B800-850 complex of rhodopseudomonas palustris.
WELTE W.

**8-01- 85** **DB21**
Neutron low resolution study of photoreaction center from wild type rhodobacter sphaeroides by contrast variation.
ARNOUX B., DUCRUIX A., REISS-HUSSON F., ROTH M.

**8-02-133** **D11**
Study of the molecular mechanism of translocation in protein biosynthesis.
SERDYUK I.N., SPIRIN A.S., BARANOV V.I.

**8-02-135** **D11**
Structure and function of E. Coli ribosomes.
NIERHAUS K.H., MAY R.P., NOWOTNY V., NOWOTNY P.

**8-02-138** **D11**
Interaction of E.Coli seryl-tRNA synthetase with its substrates.
LEBERMAN R., HAERTLEIN M., ZACCAI G.

**8-02-139** **D11**
Structure and function of E. coli ribosomes.
NIERHAUS K.H., MAY R.P., NOWOTNY V., NOWOTNY P.

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**8-02-139** **D11**
Structure and function of E. coli ribosomes.
NOWOTNY V., NOWOTNY P., NIERHAUS K.H., MAY R.P.

***8-02-128** **D11**
Interactions of DNA and viral connectors.
CARRASCOSA J.L., MERRANZ L.

**8-03-134** **D11, D17**
Studies of chromatin structure.
BALDWIN J.P., WOOD M.J., FOWLER A.G., GOLDBERG M.W., BRADBURY E.M., GODFREY J.

**8-03-135** **D11**
The DNA gyrase-DNA complex.
MAXWELL A., GELLERT M., O'DEA M.H., KRUEGER S., WLODAWER A. LANGOWSKI J., ZACCAI G.

**8-03-136** **D19**
The location of water molecules around the A-form of the DNA double helix.
FULLER W., PIGRAM W.J., MAHENDRASINGAM A., FORSYTH V.T., LANGAN P., MASON S.

**8-03-138** **D11**
Translocation of RNA polymerase.
HEUMANN H., LEDERER H., MAY R.

**8-03-142** **D11**
Cooperativity in heterologous tet repressor operator systems.
HILLEN W., TOVAR K., HEUMANN H., LEDERER H., MAY R.

**8-03-143** **D19**
The location of water molecules around the right- and left-handed forms of the poly d(G-C).poly d(G-C).
FULLER W., PIGRAM W.J., MAHENDRASINGAM A., FORSYTH V.T., LANGAN P., DENNY R., MASON S.

**8-03-144** **D11, D17**
Neutron scattering of the Pf1 nucleo-protein complex in solution.
KNEALE G.G., PLYTE S.E.

**8-04-116** **IN10, IN5**
Local lipid dynamics in bilayer membranes.
RICHTER D., KNOLL W., SACKMANN E., PFEIFFER W., HENKEL T.

**8-04-132** **IN5**
Local lipid dynamics in bilayer membranes.
PFEIFFER W., RICHTER D., KNOLL W., SACKMANN E.

**8-05-130** **D16**
Differences in the water and hydrogen distribution between the ground state and the M-intermediate of bacteriorhodopsin (BR).
BUELDT G., DENCHER N. A., DRESSELHAUS D., ZACCAI G.

**8-05-166** **D11**
Low-resolution structure of protein-SDS complexes.
KIRSCHNER K., LUNDAHL P., IBEL K., MAY R.

**8-05-170** **D17**
Solutions of detergent micelles of relevance for membrane protein crystallization.
WELTE W.

**8-05-171** **D11**
Studies of detergent-solubilized membrane proteins. Molecular weight and shape determination.
WELTE W.

**8-05-173** **D17**
A study of aqueous detergent solutions for membrane protein crystallization.
WELTE W.

**8-05-174** **D11**
Studies of detergent solubilized membrane proteins, molecular weight and shape determination.
WELTE W., LEONHARD M.

**8-05-175** **D16**
Transmembrane location of retinal in the purple membrane.
HEYN M.P., GRZESIEK S., HAUB T., OTTO H.

**8-05-176** **D11, D17**
Molecular architecture of E. Coli H-ATPase (FoF1). IV.
ALTENDORF K.H., DECKERS-HEBESTREIT G., IBEL K., SCHMID R., STEFFENS K.

**8-05-178** **D11**
Membrane protein complexes in contrast matched vesicles.
HUNT J., FLANAGAN J., MCCREA P., ZACCAI G., ENGELMAN D.

**8-05-179** **D11**
Neutron scattering study of the beef heart $F_1F_0$ ATpase complex.
DUPUIS A., WADE R., ZACCAI G.

**8-05-180** **D16**
Neutron diffraction studies of the orientation of phospholipid headgroups at the surface of lipid bilayers.
BRADSHAW J., EDENBOROUGH M., SIZER P., WATTS A.

**8-06- 63** **D11**
Morphology of a tymovirus: TYMV and its mechanism of decapsidation.
WITZ J., ADRIAN M., TIMMINS P.A.

**8-06- 64** **D11**
Structural properties of virus like particles (VLPs) produced in yeast.
KINGSMAN A., ADAMS S., BURNS N., PARDON J.

**8-06- 65** **D11**
Additional solution scattering studies of virus-like particles isolated from yeast.
KINGSMAN A., RICHARDSON M., BURNS N., PARDON J.

**8-07-121** see **8-07-135**

**8-07-132** **D11**
Structure of human complement serine proteinase Clr and its catalytic domains.
AUDE C., ZACCAI G., ARLAUD G., COLOMB M.

**8-07-135, 8-07-121** **D11**
Experimental testing of the new isomorphous replacement method in small-angle neutron scattering.
SERDYUK I.N., SPIRIN A.S., PAVLOV YU.M., LEBERMAN R., BARANOV V.I.

**8-09- 11** **D11**
Disintegration of turnip yellow mosaic virus under high hydrostatic pressure I.
LECHNER M.D., GOLDBECK A., NORDMEIER E., WITZ J., IBEL K.

# College 9

## Chemistry.

### 9a-Small molecules

**9-03-451** **D17, D11**
Backbone conformation and local packing of side chain liquid crystal polymers.
RICHARDSON R.M., ROSER S.J., LEADBETTER A.J., ETHERINGTON G., GRAY G.W.

**9-03-495** **IN6, D16**
Gaz solubilization in fluorocarbons.
RAVEY J.C., SERRATRICE G.

**9-03-524** **IN6**
Sulfuric acid solutions under an electric field.
CAVAGNAT D., LASSEGUES J.C.

**9-03-526** **IN6**
Gas solubilization in fluorocarbons.
RAVEY J.C., SERRATRICE G.

**9-03-527** **IN5**
Diffusive motion of $NH_4^+$ in $NH_4MnCl_3$.
BARTOLOME J., RUBIN J., KEARLEY G.J., MAGERL A., GRAAF DE L.

**9-03-547** **IN10**
Low temperature phases of acetone.
CLOUGH S.

**9-03-548** **IN10**
The E-E tunnelling transitions in acetylacetone.
HORSEWILL A.J., AIBOUT A., MAHLING S.

**9-03-549** **IN5**
Hindered rotations of methane in argon-nitrogen matrices.
KNOEZINGER E., LANGEL W., SCHULLER W., PRAGER M.

**9-03-549** **IN5, IN3**
Hindered rotations of methane in argon-nitrogen matrices.
SCHULLER W., PRAGER M., LANGEL W.

**9-03-550** **IN13**
Dynamics of proton transfer in fatty acids.
HAYASHI S., HEIDEMANN A.

**9-03-551** **IN10**
The influence of the nature of the chemical bonding on the rotational tunnelling in methyl tin compounds $(CH_3)_3SnNa$.
PRAGER M., WEISS A., ZHANG D.

**9-03-552** **IN10**
Intramolecular interaction in methyl tin compounds $(CH_3)_3Sn\text{-}Sn(CH_3)_3$.
PRAGER M., WEISS A., ZHANG D.

**9-03-553** **IN5**
Origin of the rotational barrier of coordinated molecular hydrogen in metal complexes.
ECKERT J., KUBAS G.J.

**9-03-554** **IN5**
High resolution neutron scattering study of rotational excitations in mixed $CH_4/CD_4$ systems.
PRAGER M., PRESS W.

**9-03-556** **IN5**
The $-NH_2$ inversion tunneling in urea isolated in an argon matrix.
JAYASOORIYA U.A., GRINTER R.

**9-03-560** **IN5**
Dynamics of Complex-Cations in Hydroxysodalites $Na_{6+x}(AlSiO_4)_6.(OH)_x.nH_2O$.
BUEHRER W., ANDERSON I., FELSCHE J.

**9-03-561** **IN5**
Translational diffusion in $H_2SO_4/H_20$.
CAVAGNAT D., LASSEGUES J.C.

***9-03-568** **IN10**
Coupled methylrotators in $(CH_3)_2SnCl_2$.
STANISLAWSKI J., PRAGER M., MAGERL A.

**9-03-580** **IN13, IN8, D1B**
Correlated longitudinal motions in oligotetrafluoroethylenes.
ALBRECHT T., STEINER R., STROBL G., STUEHN B.

**9-03-583** **IN13**
$HNO_3$ intercalated in graphite.
SIMON C., ROSENMAN I., BATALLAN F., ROGERIE J.

**9-03-586** **IN10**
Lithium diffusion in stage-2. Lithium-graphite intercalation compounds.
KIM H.J., FISCHER J.E., MAGERL A.

**9-04-348** **IN6**
Low-frequency librational motions in the plastic phase pivalic acid.
LONGUEVILLE W., BEE M.

**9-04-390** **IN13, IN6**
Water dynamics in tungsten trioxide hydrates $WO_3$, $xH_20$ with x= 1, 2, 1/3.
LASSEGUES J.C., DESBAT B., GERAND B., FIGLARZ M.

**9-04-391** **IN4, IN6, IN13**
Librational energy levels and rotational dynamics of $H_20$ in sodium perchlorate monohydrate.
STRAUSS H.L., LASSEGUES J.C.

**9-04-396** **IN6**
Influence of the local order on the densities of states in two amorphous phases of liquid crystal MBBA.
FOURET R., BEE M.

**9-04-399** **IN 3**
Collective methyl torsion modes in alkanes.
CLOUGH S., KEMP A.

**9-04-401** **IN5**
Dynamics of flip-flop hydrogen bonding disorder in β-cyclodextrin.$12H_2O$.
SAENGER W., LECHNER R.E., STEINER T.

**9-04-402** **IN4**
Relaxation processes in the magnetic exchange transitions in mixed-valence and mixed-metal systems.
JAYASOORIYA U.A., CANNON R.D., BOLLEN S.K., CHAI SAARD N., WHITE R.P.

**9-04-403** **IN6**
Influence of the local order on the density of states in amorphous phases of liquid crystal MBBA.
FOURET R., GORS C., BEE M., DEROLLEZ P.

**9-04-404** **IN3**
Methyl torsion in a lamellar compound.
VENIEN J.P., CLOUGH S.

**9-06-546** **IN10**
Quasielastic neutron scattering of third stage $HNO_3$ intercalated in graphite.
BATALLAN F., SIMON C., ROSENMAN I., MAGERL A.

**9-06-549** **IN5**
Dynamics of methane in ZSM-5 zeolite.
JOBIC H., BEE M.

**9-06-552** **D1B**
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