

# ANNUAL REPORT 85

INSTITUT MAX VON LAUE - PAUL LANGEVIN - GRENOBLE - FRANCE



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# Applications for the use of the 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

156 X

38042 Grenoble Cedex

France

Tel. 76 48 72 44 B. Maier

76 48 71 79 G. Briggs

76 48 70 41 K. Mayer-Jenkins (Secretary)

76 48 70 82 F. Cook (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 three 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 scientist considers that this is the case, he should get in touch with the Scientific Secretary.

## Other publications available

Neutron Research Facilities at the ILL High Flux Reactor, Edition 1983.

Information and Regulations for Reactor Users, Long-Term Visitors and New Scientific Staff, Edition March 1984

both available from SCAPRO

Experimental Reports and Theory College Activities 1985 available from the ILL Library.

## Front Cover

The UCN-turbine at the niveau D of the reactor. The neutron guide extracting very cold neutrons from the new vertical Cold Source can be seen on the left.

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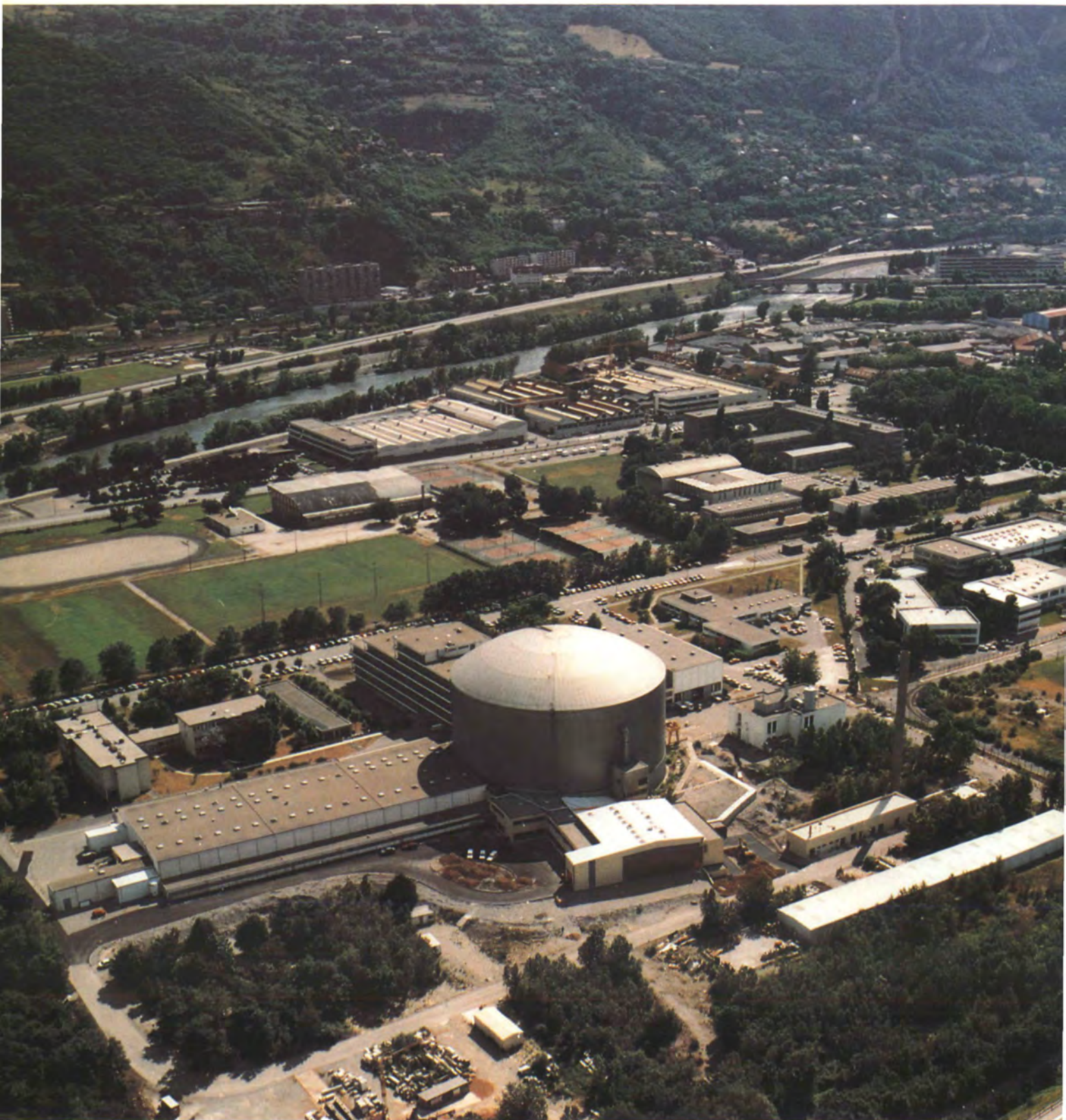
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*The Institut Max von Laue - Paul Langevin*

# The Institut Max von Laue - Paul Langevin

**T**he 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 Institut 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 1, 1973 the United Kingdom joined the Institut 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 at least 1992 and beyond unless two years' notice of termination has been given.

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 Institut 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 central facility 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.

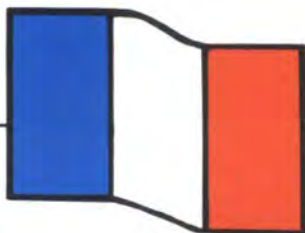
Designing and operating instruments and helping the visiting users to carry out their experiments is thus the principal task of the Institut's own scientists. The experimental use of the instruments by ILL staff is subject to the same approval system as their use by external teams.

# External Organisation of the Institut Laue-Langevin 1985

## Associates of the Institut



Science and Engineering  
Research Council (SERC)



Commissariat à l'Énergie  
Atomique (CEA)  
Centre National de la Recherche  
Scientifique (CNRS)



Kernforschungszentrum  
Karlsruhe (KFK)

## Steering Committee (at its last meeting)

Atkinson (SERC)	Cribier (CEA)	Blaesing (BMFT)
Clark (SERC)	Horowitz (CEA)	Hofbauer (BMF)
Leadbetter (SERC)	Lehmann (CNRS)	Klose (KFK) (chairman)
Otewill (Univ. of Bristol)	Miquel (CNRS)	Simon (Max-Planck-Ges.)

## Audit Commission

Pettet	Polderman	Binder
Millington	Racine	Riess

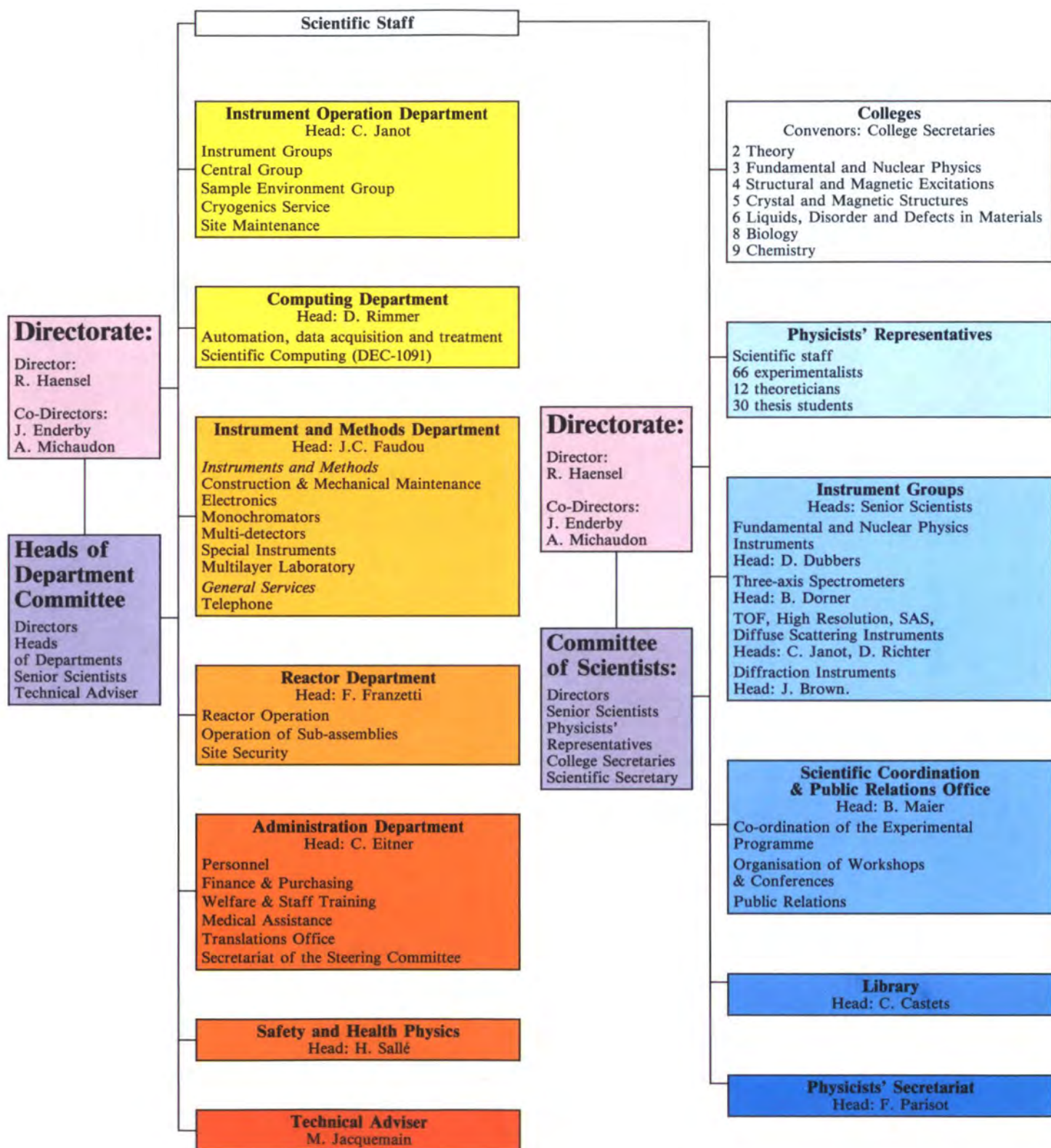
## Scientific Council (at its last meeting)

Blanquet — CNRS Palaiseau	Haensel — ILL (chairman)	Press — Univ. Kiel
Comes — Orsay	Hahn — TH Aachen	Rainford — Univ. Southampton
Cowley — Univ. Edinburgh	Maier — ILL	Rossat-Mignod — CEN Grenoble
Dianoux — ILL	Marezio — CNRS Grenoble	Scherm — PTB Braunschweig
Drifford — CEN Saclay	Michaudon — ILL	Schmatz — KFK Karlsruhe
Enderby — ILL	Nierhaus — MPI Berlin	Schult — KFA Jülich
Franck — Univ. Karlsruhe	Otewill — Univ. Bristol	Smith — Unilever
Fuller — Univ. Keele	Pendry — Imperial College	Stirling — ILL
Gläser — TU München	Perez y Jorba — Orsay	Thomas — Univ. Oxford
Gönnenwein — Univ. Tübingen	Petroff — LURE	Zinn — KFA Jülich

## Subcommittees of the Scientific Council

Fundamental and Nuclear Physics	Structural and Magnetic Excitations	Crystal and Magnetic Structures		Liquids, Disorders and Defects in Materials	Biochemistry	Chemistry		Instruments
						Spectroscopy Surfaces & Mesophases	Colloids & Polymers	
Barloutant Gelletly <i>Gönnenwein</i> Lefort Lieb Pendlebury	Boucher Edwards Hennion Hutchings Rainford <i>Schmatz</i> Steiner Vallade Zeyher	Bronger Cheetham Finney Fischer <i>Marezio</i> Pouget	Coles Dachs Day Flouquet Fuess Györfly Plumier <i>Zinn</i>	<i>Comes</i> Dupuy Durand Gaskell Gläser Mc Donald Peisl Stewart Suck	Chabre Finch Giege Henderson Jacrot Janin Miller <i>Nierhaus</i> Philipson Saenger Schulz	Clough Conard Forstmann Hueller Lassegues <i>Thomas</i>	Cabane Cotton Hoffmann Kilian <i>Otewill</i> Picot Richards	Alefeld Bergere Charpak Pepy Reichardt <i>Scherm</i> Stewart Steyerl Taylor Williams

# General Organigramme



# Visits and Events in 1985



*Prof. Haensel started his tenure as Director on 1 April 1985. In the picture he (on the right) is seen with the British Assistant Director, Prof. Enderby, who commenced his work at the ILL on 1 August 1985.*



*After a reactor shut-down of 11 months the reactor staff and the scientists were impatiently waiting for the first neutrons in 1985. On 26 August at about 17<sup>00</sup>h the reactor became critical again. In the picture from left to right F. Franzetti, J.M. Astruc, P. Ageron and O. Schärpf enthusiastically discussed the first flux results obtained with the new cold source.*

*Lord Lucas of Chilworth, British Minister of State for Trade and Industry, visited the ILL on 13 November 1985. On his tour through the ILL he was guided by the Director, Prof. Haensel and the British Assistant Director, Prof. Enderby. In the picture Lord Lucas (third from left) receives explanations by Prof. Enderby about the operation of the HFR.*



# Bericht des Direktors

O

bwohl der Reaktor die überwiegende Zeit des Jahres stillstand und erst am 3. September 1985 seinen Routinebetrieb wieder aufnahm, war es kein langweiliges Jahr für das ILL; im Gegenteil: es war spannend vom ersten bis zum letzten Tag.

## Modernisierungsprogramm

Am 31. Dezember 1985 ging das Modernisierungsprogramm 1979-85 (deuxième souffle) offiziell zu Ende. Während des 11 Monate andauernden Reaktorhalts (3.10.84 - 2.9.85) wurden die komplizierten Arbeiten, die im Rahmen dieses Modernisierungsprogramms am Reaktor selbst durchzuführen waren, erfolgreich abgeschlossen. Mit Auswechseln der Strahlrohre H1/H2 und des durchgehenden Strahlrohrs H6/H7 sind nun alle Einheiten im Innern des Reaktors ausgetauscht worden. Der beschädigte Schwerwasseransaugstutzen wurde mittels eines ausgetüftelten Elektroerosionsverfahrens unter Wasser abgetragen. Die vertikale kalte Quelle wurde durch eine neue ersetzt, die in den Strahlrohren für kalte Neutronen die beachtliche Flusssteigerung um 60 % bewirkte. Alle diese komplizierten Arbeiten sind von der Reaktorabteilung selbst geplant und durchgeführt worden, sie verliefen ohne grössere Zwischenfälle und konnten streng nach Zeitplan beendet werden. Im Rahmen des Modernisierungsprogramms wurde auch eine Zahl von Instrumenten umgebaut, erweitert und verbessert; zum grössten Teil sind diese Arbeiten beendet, ein kleiner Teil der Instrumente befindet sich noch bei abschliessenden Tests.

In der Reaktorhalle wurden die Instrumente D2 und D8 durch ein Dreifachinstrument D2B, D19 und D20 ersetzt. D9 wurde um 3,5 m näher an den Reaktorkern verlegt. Durch Ausdehnung des Monochromators wurde für IN8 der Fluss um einen Faktor 1,5 verbessert. Das neue Gerät IN20 löst IN2 ab.

In der Neutronenleiterhalle wurde die Kleinwinkelapparatur D11C umgebaut. Die Verschiebbarkeit des Multidetektors unter Vakuum wird einen sehr raschen Wechsel der Instrumentenparameter und damit eine wesentliche Verkürzung der Umrüstzeiten von Experiment zu Experiment erlauben. Die Instrumente D1A und D1B wurden in ihrer Position am Neutronenleiter vertauscht. Zwischen D7 und IN10 wurde der Neutronenleiter unterbrochen, um Platz für den Auslass für das gemeinsam mit dem Europäischen Molekularbiologie-Labor EMBL gebaute Biologie-Diffraktometer DB21 zu schaffen. Die Chopper von IN5 wurden modernisiert und laufen nach anfänglichen Schwierigkeiten jetzt einwandfrei.

Das grösste, innerhalb des Modernisierungsprogramms finanzierte Teilprojekt, die zweite (horizontale) kalte Quelle, wird planungsgemäss erst im Jahre 1986 fertiggestellt sein. Möglicherweise wird sie aber erst 1987 eingesetzt werden können, da die Instrumente für die zweite kalte Quelle wegen der vielfältigen Arbeiten am und um den Reaktor während des langen Reaktorhalts zurückgestellt werden mussten. Für die zweite kalte Quelle wurde eine Experimentierhalle gebaut, in der folgende Instrumente stehen sollen: IN10C, eine verbesserte Version des Rückstreupektrometers IN10B; IN15, ein neues Spin-Echo-Instrument, das gemeinsam vom ILL, dem BMFT, der KFA Jülich und dem HMI Berlin finanziert und gebaut wird; als weiteres Instrument soll D22 den grossen Andrang bei den Kleinwinkelapparaturen bewältigen helfen. Das vierte Instrument an der 2. kalten Quelle ist IN14, ein 3-Achsenspektrometer, welches an einem der neuen Neutronenleiter in der Reaktorhalle installiert werden wird. Als fünftes Projekt an der zweiten kalten Quelle soll das Neutron-Antineutron-Experiment untergebracht werden.

## Das wissenschaftliche Programm

Die als Folge des Modernisierungsprogramms erreichten Verbesserungen können in 3 Kategorien zusammengefasst werden. Mehrere Instrumente werden konkurrenzlos sein: Zum Beispiel das neue Spin-Echo-Spektrometer (IN15) ist für eine Energieauflösung von 0.1neV und einen minimalen Impulsübertrag  $Q$  von  $10^{-3} \text{ \AA}^{-1}$  ausgelegt. Untersuchungen über die Dynamik von Polymerlösungen, besonders am

Überschneidungspunkt von verschiedenen Diffusionsgebieten, werden mithin möglich werden. IN15 wird ausserdem die Analyse kritischen dynamischen Verhaltens nahe an magnetischen und strukturellen Phasenübergängen erlauben und die Erforschung von Spindynamik und Spinrelaxation in Spin-Systemen mit Unordnung und niedriger Dimension ermöglichen. Ein anderes neues Instrument ist DB21, ein Kleinwinkeldiffraktometer für Proteinkristallographie. Biologische Makromoleküle und Materialien bestehen oft aus einem Komplex von zwei oder mehreren getrennten chemischen Komponenten (Chromosomen, zum Beispiel, bestehen aus Proteinen und DNS). Kontrastvariation mit  $\text{H}_2\text{O}/\text{D}_2\text{O}$  Mischungen unter Anwendung von Neutronenstreuung ist eine sehr effiziente Methode zur Unterscheidung verschiedener Komponenten innerhalb eines Makromoleküls. Klassische Methoden würden sehr hohe Auflösung erfordern, was in vielen Fällen ein aufwendiges und sogar technisch unmögliches Unterfangen wäre. DB21 wurde für Untersuchungen mit kleiner Auflösung unter Heranziehung der H/D-Markierung optimiert. Die gelieferte Information wird hochauflösende Röntgenstrahlexperimente ergänzen oder sogar einen wertvollen eigenständigen Beitrag liefern.

Für eine relativ grosse Gruppe von Instrumenten, einschliesslich 3-Achsenspektrometer, Diffraktions- und Flugzeitgeräten, wurde die Effizienz um eine Grössenordnung gesteigert. An D4B, dem Diffraktometer für Flüssigkeiten und amorphen Festkörpern, können nun Experimente mit Isotopensubstitution an sehr verdünnten Systemen durchgeführt werden: zum Beispiel wurde bei sehr kleinen (3 at% Cu) Konzentrationen der von Kupfer gebildete Koordinierungskomplex in amorphem  $\text{As}_2\text{Se}_3$  untersucht. Mehrere Experimente wurden durchgeführt und zusätzliche in Quasikristallen vorgeschlagen, d.h. solchen welche icosahedrische Phasen enthalten und gewöhnlich durch rasche Verfestigung von gewissen Aluminiumlegierungen gebildet werden. Zum Beispiel ein vor kurzem an D1B durchgeführtes Experiment nutzte die negative Streulänge von Mangan und mit Hilfe von Kontrastvariation (Fe wurde mit Mn substituiert) konnte man eine direkte Information über die Al-Al-Struktur erhalten. Man plant im Augenblick, die ausgezeichneten Eigenschaften von D4B für diese Legierung und verwandte quasikristalline Systeme in der flüssigen Phase zu nutzen und somit eine mikroskopische Vorstellung von der Stabilität von icosahedrischen Phasen zu bekommen. Die neue Version von IN2, IN20, erlaubt die Durchführung von inelastischen Polarisationsexperimenten. So unterschiedliche Probleme wie die augenscheinliche Entartung von gewissen magnetischen und Gitteranregungen in USb und die Identifizierung von Magnonen in  $\text{RbFeCl}_3$  können nun am IN20 in Angriff genommen werden. Die Verbesserungen an IN1 (dem 3-Achsenspektrometer an der heissen Quelle) ermöglichten ein wichtiges Experiment mit Nickel. Die Spinwellen-Moden wurde bei ihrem Zusammentreffen mit der dichten Region von Stoner-Anregungen untersucht. Man konnte zum ersten Mal aufzeigen, dass sich die Spinwelle fortsetzt - ein Ergebnis von beträchtlicher Bedeutung für Band-Theoretiker, die sich mit grundlegenden Problemen des Ferromagnetismus in Nickel befassen. IN1 ermöglicht diese Experimente aufgrund der vergleichsweise kleinen Q-Werte, die die Untersuchung von hochenergetischen Anregungen gestatten.

Schliesslich wurden mehrere Instrumente in bescheidenerem Umfang verbessert; nichtsdestoweniger werden diese Modernisierungsmassnahmen bedeutend sein für die Erschliessung neuer Wege in der Wissenschaft. Am IN10, dem sehr populären Rückstreupektrometer, waren die wissenschaftlichen Möglichkeiten bisher immer durch den niedrigen Fluss begrenzt. Die neuen Analysatorkristalle und der erhöhte Fluss von der modifizierten kalten Quelle ergaben einen Verbesserungsfaktor von etwa 2. Dies wird zum Beispiel aufgrund der Verwendung geringerer Wasserstoffkonzentrationen wesentlich sauberere Untersuchungen der Wasserstoffdynamik in Metallen erlauben, was gleichbedeutend ist mit weniger Wechselwirkung zwischen Wasserstoffatomen. Ebenso kann der Isotopeneffekt besser charakterisiert werden, der dann entsteht, wenn Wasserstoff durch Deuterium ersetzt wird. Die verbesserte Kleinwinkelstreuanlage D11C (Ersatz für D11) hat ein wichtiges neues Kennzeichen: Die Entfer-

nung zwischen Probe und Detektor kann von 1,2m bis 36m variiert werden. Dies bedeutet, dass ohne die Probe oder ihre Umgebung zu stören, Q-Regionen von  $10^{-3}$  bis  $10^{-1} \text{ \AA}^{-1}$ , die eine detaillierte Untersuchung rechtfertigen, überstrichen werden können.

Das Modernisierungsprogramm hat auch im Bereich Kern- und Grundlagenphysik einige Neuerungen gebracht. Die auffallendste Neuerung war die Installation der neuen ultrakalten Neutronenquelle oberhalb des Reaktors. Diese neue Quelle liefert mehr ultrakalte Neutronen als alle anderen bisherigen Quellen (mehr als 2 Grössenordnungen). Im Laufe des Jahres 1986 sollen an dieser Quelle ein halbes Dutzend Instrumente installiert werden.

In der Grundlagenphysik gingen die Bestrebungen weiter, endlich die Grösse der schwachen Kopplungskonstanten zu erfassen. Mit dem Neutronenzerfallspektrometer PERKEO gelang es, das Verhältnis der Kopplungskonstanten zu verschiedenen Anteilen der schwachen Wechselwirkung genau zu bestimmen. Des weiteren wurde ein breites Programm angelegt, endlich die Lebensdauer des freien Neutrons genauer zu messen. Die Lebensdauer des Neutrons liefert bisher die einzige Möglichkeit, die globale Stärke der schwachen Wechselwirkung genau zu bestimmen. Daraus ergeben sich alle anderen schwachen Wirkungsquerschnitte und Lebensdauern, die sonst nicht direkt messbar sind, und die in der Kosmologie, der Astrophysik und der Physik der Sonne eine grosse Rolle spielen. In diesem Zusammenhang ist zu erwähnen, dass es gelungen ist, Neutronen in einer Flasche mit flüssigen Wänden über sehr lange Zeiten zu speichern.

In der Kernspaltungsgruppe konnten die Untersuchungen spaltbarer Systeme ausgedehnt werden auf weitere Aktinide wie Californium und Neptunium. Dadurch ergeben sich interessante Vergleiche mit den Ergebnissen aus den bisherigen Messungen aus der Spaltung am Uran. Die Arbeiten zur sogenannten kalten Spaltung von Kernen wurden fortgesetzt.

Zur Strukturuntersuchung an Kernen stehen am ILL mit den Instrumenten GAMS und BILL Apparate zur Verfügung, die die weltweit beste Auflösung sowohl für Röntgen- und Gammastrahlung als auch für Elektronenstrahlung bieten. An ihnen wurden die Messungen zur Bestätigung des interaktiven Bosonenmodells fortgeführt. Insbesondere gelang es, elektrische Monopolübergänge zu identifizieren. Mit der Installation des neuen Instruments GAMS 4 gelang es, die Auflösung für Gammastrahlen nochmals um mehrere Grössenordnungen zu verbessern.

Aus dem Bereich der Kernphysikgruppe ging auch die Entwicklung eines neuen Typs von Detektoren hervor, der erstmals am Spaltproduktspektrometer Cosi fan tutte eingesetzt wurde, und mit dem es möglich ist, Neutronen und andere Teilchen gleichzeitig mit sehr hoher Ortsauflösung und sehr hoher Energieauflösung grossflächig zu messen. Es ist abzusehen, dass diese neuen Detektoren in der gesamten Neutronenphysik als auch in der Kern- und Elementarteilchenphysik eine grosse Rolle spielen werden.

## Finanzielle Situation

Schwierigkeiten in den Forschungsbudgets einiger Mitgliedsländer sind auch am ILL nicht spurlos vorübergegangen, obwohl die Auswirkungen sicher geringer waren als an manchen nationalen Instituten.

Um die Budgetentwicklung des ILL besser bewerten zu können, haben die Gesellschafter eine Expertengruppe eingesetzt, die 'den Gesellschaftern Hinweise auf Einsparungsmöglichkeiten und Empfehlungen für Prioritäten im Programm des ILL' geben sollten. Die Expertengruppe beriet zwei mögliche Budgetrahmen und zeigte auf, wo nach ihrer Ansicht der untere Budgetansatz liegen müsse, wenn das Institut nicht ersten Schaden nehmen sollte. Die Gesellschafter haben für 1986 dann einen Budgetansatz gebilligt, der erfreulicherweise über dieser Untergrenze lag. Angesichts der grossen Schwierigkeiten, die 17,5 Stellen des Modernisierungsprogramms wieder abzubauen, haben die Experten geraten, für die nächsten Jahre von weiteren Stellenreduzierungen abzusehen. Natürlich hat ein Institut nach 20 Jahren durch diese und jene Massnahme eine Struktur, die nicht genau der entspricht, die man sich für ein neugegründetes

Institut dieser Grösse und mit dieser Aufgabenstellung vorstellen würde. Das Annähern an die 'Idealstruktur' kann nur langfristig erfolgen, wobei natürlich auch das Modell der 'Idealstruktur' langfristigen Änderungen unterworfen ist.

## Personalangelegenheiten

Am 1. April fand ein grosser Wachwechsel im Direktorium statt: B.E.F. Fender schied nach 5-jähriger Tätigkeit aus dem Institut aus, um seinen Dienst als Vizekanzler der Universität Keele (Grossbritannien) anzutreten. Sein Nachfolger kommt aus Kiel (Bundesrepublik Deutschland). Der stellvertretende deutsche Direktor, Prof. H. Ruppertsberg, schied nach zweieinhalbjähriger Tätigkeit aus und ging an seine Heimatuniversität Saarbrücken zurück. Ihm folgte nach viermonatiger Pause am 1. August 1985 als neuer stellvertretender britischer Direktor Prof. J.E. Enderby von der Universität Bristol. A. Michaudon sorgte für Kontinuität in dieser Zeit.

Nachdem am 23. Mai 1985 P. Nozières in Jerusalem den Wolf-Preis 1985 für Physik entgegennehmen konnte, fiel inzwischen ein weiterer Preis an ein (ehemaliges) ILL-Mitglied: F. Mezei, bis 31.7.1984 am ILL, seither ordentlicher Professor an der Technischen Universität Berlin, erhielt den Hewlett Packard Preis 1986 für seine 'fundamentalen Arbeiten zur Entwicklung der Spin-Echo-Methode', die er am ILL so erfolgreich entwickelt hat. Der Preis wird ihm während der EPS-Tagung in Stockholm Ende März 1986 überreicht werden.

Als Nachfolger von W. Press, der einen Ruf als ordentlicher Professor an die Universität Kiel annahm, hat D. Richter von der Kernforschungsanlage Jülich seine Funktion als Senior Scientist übernommen. Ähnliches gilt für D. Dubbers (Universität Heidelberg), der F. Gönnewein (Universität Tübingen) als Senior Scientist für den Bereich Kern- und Elementarteilchenphysik nachfolgte.

Neben den namentlich genannten ist aber das ILL auch auf seine übrigen Mitarbeiter stolz. Die vielfältigen Erfolge bei den Arbeiten am und um den Reaktor, an den Instrumenten, bei den Rechenanlagen, in der Verwaltung zeigten den unermüdlichen Einsatz und die Begeisterung der Mitarbeiter, die auch nach 13 Jahren Betrieb 'ihr' ILL wie am ersten Tag lieben. Diese Liebe zum ILL zeigte sich nicht zuletzt an einigen heftigen Reaktionen auf notwendige Sparmassnahmen der Direktion am Ende des Jahres 1985.

## Zukunftspläne

Nachdem das ILL im Frühjahr 1984 vorgeschlagen hatte, Grenoble zum Standort für die Europäische Synchrotron-Strahlungs-Quelle (ESRF) zu machen und Oktober 1984 die französische und deutsche Regierung sich diesen Vorschlag zu eigen gemacht hatten, wurden während des Jahres 1985 in Grenoble die ersten Schritte zur Gründung des neuen Instituts getan: Frankreich, die Bundesrepublik Deutschland, Italien und Grossbritannien haben sich fest verpflichtet, das neue Projekt zu finanzieren, einige andere Länder werden sich hoffentlich noch anschliessen. Die Träume meines Vorgängers reifen also.

Für das ILL selbst wird ebenfalls eine erweiterte Mitgliedschaft angestrebt. Eine lockere Anbindung anderer Länder im kommenden Jahr könnte ein erster Schritt zu einer Konstellation sein, die nach 1992 ein ILL mit mehr als 3 Mitgliedsländern zur Folge haben könnte.

## POSTSCRIPTUM

Es sieht so aus, als ob der erste Jahresbericht des neuen Direktors auch schon sein letzter ist. Er folgte dem Ruf des neuen Nachbarn ESRF. So bleibt mir zum Schluss nur übrig, allen Mitarbeitern des ILL für ein Jahr zu danken, das ich vom ersten bis zum letzten Tag genossen habe.

R. Haensel

# Director's Report

**A**lthough the reactor was shut down for the majority of the year and only resumed routine operation on 3 September 1985, this was not a dull year for the ILL; on the contrary it was exciting from beginning to end.

## Modernisation Programme

On 31 December 1985 the modernisation programme 1979-85 (also known as the 'deuxième souffle') officially ended. During the 11-month reactor shut-down (3.10.84 - 2.9.85) the complex tasks to be carried out on the reactor itself under this modernisation programme were successfully completed. With the replacement of beam tubes H1-H2 and the through-going beam tube H6-H7 all the internal reactor units have now been renewed. The damaged heavy water collector was removed under water by means of a sophisticated electro-erosion process. The vertical cold source was replaced by a new source, which gives a considerable increase in flux of 60 % in the cold neutron beam tubes. All these complex projects were planned and carried out by the reactor department itself, and were implemented without any serious incident and completed precisely to schedule. Various instruments were also rebuilt, extended and improved during the modernisation programme. This work is largely completed, but a few of the instruments are still undergoing final tests. In the reactor hall the instruments D2 and D8 have been replaced by a group of three instruments D2B, D19 and D20. D9 was moved 3.5 m closer to the reactor core. The flux for IN8 was improved by a factor 1.5 by increasing the height of the monochromator. The new instrument IN20 replaces IN2.

In the neutron guide hall the small-angle instrument D11 was rebuilt. The automatic displacement of the multidetector under vacuum will permit rapid changes in instrument parameters and result in a considerable shortening of the change-over times from one experiment to another. The positions of the instruments D1A and D1B on the guide have been reversed. Between D7 and IN10 the neutron guide was interrupted to make room for the biology diffractometer DB21 constructed jointly with the European Molecular Biology Laboratory. The choppers on IN5 have been modernised and after some initial difficulties are now running well.

The largest project financed under the modernisation programme, the second (horizontal) cold source, will be completed as planned in 1986. However it may only be possible to install it in 1987 as the instruments for the second cold source had to be postponed because of the variety of work on and around the reactor.

A new guide hall was built for the second cold source, in which the following instruments are to be installed: IN10C, an improved version of the backscattering spectrometer IN10B; IN15, a new spin-echo instrument, which will be financed and built jointly by the ILL, BMFT, KFA Jülich and HMI Berlin and an additional instrument D22, which should help to deal with the considerable pressure on the small-angle instruments. The fourth instrument on the second cold source is IN14, a triple-axis spectrometer which will be installed on the neutron guide but actually located in the reactor experimental hall. The neutron-antineutron experiment is to be installed as a fifth project on the second cold source.

## Scientific Programme

Improvements which followed the instrument modernisation programme can be described under three main categories. First there are a group of instruments which, when fully operational, will have no present-day equivalent. For example, the new spin-echo spectrometer (IN15) is designed to have an energy resolution of 0.1 neV and a minimum Q (momentum transfer) value of  $10^{-3} \text{ \AA}^{-1}$ . Studies of the dynamics of polymer solutions, especially at the cross-over between various diffusion regions, will now be possible. IN15 can be used to study critical dynamics adjacent to magnetic and structural phase transitions and for research into spin dynamics and spin relaxation in

disordered and low-dimensional spin systems. Another new instrument, DB21, is the small angle diffractometer for protein crystallography. Biological macromolecules and materials often exist as a complex of two or more distinct chemical species (chromosomes, for example, consist of protein and DNA). Contrast variation with  $\text{H}_2\text{O}/\text{D}_2\text{O}$  mixtures using neutron diffraction is a very effective method for distinguishing different components within a macromolecule. Classical methods would require very high resolution, which in many cases is a formidable or even technically impossible undertaking. DB21 has been optimised for low resolution studies involving H/D labelling and the information provided will complement high resolution X-ray studies or indeed make a valuable contribution in its own right.

There is a large group of instruments, including triple-axis spectrometers, diffraction and time-of-flight machines, where the performance has improved by an order of magnitude. On D4B, the liquids and amorphous solids diffractometer, isotopic substitution experiments can now be performed on very dilute systems: for example, the co-ordination complex formed by Cu in amorphous  $\text{As}_2\text{Se}_3$  has been investigated at very low (3 at% Cu) concentration. Several experiments have been performed and additional ones suggested in respect of quasicrystals, i.e. those which contain icosahedral phases and which are usually formed by the rapid solidification of certain aluminium alloys. On D1B, for example, a recent experiment exploited the negative scattering length of Mn and, by contrast variation (Fe being substituted for Mn), allowed information about the Al-Al structure to be obtained directly. It is now planned to exploit the excellent performance of D4B to look at this alloy and related quasicrystalline forming systems in the liquid phases and so build up a microscopic understanding of the stability of the icosahedral phase. The new version of IN2, IN20, with its very high flux allows inelastic polarisation experiments to be performed. Problems as disparate as the apparent degeneracy of certain magnetic and lattice excitations in USb and the identification of magnons in  $\text{RbFeCl}_3$  are readily attacked on IN20. After improvements to IN1 (the hot-source triple-axis spectrometer) an important experiment on nickel was performed. The spin wave modes were investigated as they encountered the dense region of Stoner excitations. It was shown for the first time that the spin wave continues to propagate - a result of considerable significance to band theorists studying the fundamental problems of the ferromagnetism in nickel. These experiments are possible because IN1 enables high energy excitations to be studied at comparatively small values of Q.

Finally, several instruments were improved in a more modest way, but nevertheless will allow significant new science to be performed. For example, the science possible on the very popular back-scattering instrument IN10 has always been limited by the low flux. The new analyser crystals and the increased flux from the modified cold source have led to an improvement of about a factor of 2. This will, for example, allow much cleaner studies of the dynamics of hydrogen in metals to be investigated because lower hydrogen concentrations can be used which means that there is less hydrogen-hydrogen interaction. Similarly, the isotope effect which occurs when hydrogen is replaced by deuterium can also be better characterised. The improved small angle scattering instrument D11C (replacing D11) has an important new facility: the distance between the sample and the detector can be varied from 1.2 m to 36 m. This means that, without disturbing the sample or its environment, regions of Q in the range  $10^{-3}$  to  $10^{-1} \text{ \AA}^{-1}$  which warrant detailed study, can be readily investigated.

The modernization programme has also brought some innovations in the field of nuclear and fundamental physics. The most striking achievement was the installation of the new ultra-cold neutron facility which has an increase of more than two orders of magnitude over other existing sources. During 1986 some six instruments will be fed by this facility.

In fundamental physics, efforts continued to finally obtain information on the size of the weak coupling constants. On PERKEO, the

neutron decay spectrometer, it was possible to determine the ratio of coupling constants of the various parts of the weak interaction. In addition, an important programme was initiated to increase the accuracy of the free neutron life-time. This life-time is currently the only possibility to measure the global force of the weak interaction with high precision. From that one might derive all other weak cross-sections and life-times, which are otherwise not directly measurable, and which play a major rôle in cosmology, astrophysics and solar physics. In this context it is notable, that one has succeeded in storing neutrons in bottles with liquid walls for very long times.

In fission physics the investigation of fissionable systems was extended to actinides such as californium and neptunium. From that one can draw interesting comparisons with the results from the measurement of uranium. The work on the so-called cold fission of nuclei was continued.

For nuclear structure investigations the instruments GAMS and BILL have the best resolution worldwide for X- and  $\gamma$ -rays as well as for electrons. There, the investigation on the confirmation of the interacting boson model was continued. In particular one has succeeded in identifying electric monopole transitions. On GAMS4, a new  $\gamma$ -ray spectrometer, the resolution for  $\gamma$ -rays was improved by several orders of magnitude.

A spin-off from the activity of the nuclear physics group was the development of a new type of detector, which has been used for the first time at the fission product spectrometer *Cosi fan tutte*. This detector permits a large area detection of neutrons and other particles simultaneously with high position and energy resolution. One can assume, that this new kind of detector will play a major rôle in the whole area of neutron physics as well as in nuclear and fundamental physics.

## Financial Situation

Difficulties in the budgets for science in some member countries have not left ILL untouched, although the effects have certainly been less than for some laboratories and universities in the member countries.

To permit a better evaluation of the ILL budget trends, the Associates set up a group of experts, to 'advise the Associates on possible economies and recommend priorities in the ILL programme'. The group of experts discussed two possible outline budgets and indicated where they considered the lower budget limit should lie, if the Institut were not to suffer serious damage. The Associates then approved a budget figure for 1986, which was fortunately above this lower limit. In view of the considerable difficulties in eliminating the 17.5 posts under the modernisation programme, the experts advised that there should be no additional staff complement reductions over the next few years. It is clear that after twenty years, as a result of various measures, an institute has a structure which does not correspond exactly to what one would envisage for a newly founded institute of similar size and functions. The approximation to the 'ideal structure' can only be a long-term process, during which the model of the ideal structure is of course also subject to long-term modifications.

## Personnel

On 1 April there was a major change of directors: B.E.F. Fender left the Institut after five years here to devote himself to his duties as a Vice-Chancellor of the University of Keele (England). His successor comes from Kiel (Germany). The German assistant director, Professor H. Ruppertsberg, left after two and a half years and returned to his home university of Saarbrücken. After an interval of four months he was succeeded on 1 August 1985 by Professor J.E. Enderby from the University of Bristol as British assistant director. The continuity was maintained during that transition period by A. Michaudon.

Following the presentation of the 1985 Wolf Prize for Physics on 23 May to P. Nozières in Jerusalem, a further prize has been awarded to a (former) ILL scientist, F. Mezei who was at ILL until 31.7.84 and is now a professor at the Technical University in Berlin; he was awarded the 1986 Hewlett Packard Prize for his 'fundamental work on the development of the spin-echo method' which he developed so successfully at the ILL. The presentation of the prize takes place during the EPS meeting in Stockholm at the end of March 1986.

W. Press, who accepted a post as professor at the University of Kiel, was succeeded by D. Richter from the Kernforschungsanlage Jülich as Senior Scientist. The same applies to D. Dubbers (University of Heidelberg) who succeeded F. Gönnerwein (Tübingen) as Senior Scientist for the Nuclear and Elementary Particle Physics area.

In addition to those mentioned by name, the ILL is also proud of its other staff. The numerous successes in work on and around the reactor, on instruments, on computers and in the administration illustrate the tireless dedication and enthusiasm of their staff who are still as fond of 'their' ILL after 13 years operation as on the first day. This feeling for the ILL showed itself not least in some strong reactions to the ILL Management's necessary economy measures at the end of 1985.

## Future Plans

Following the ILL proposal in spring 1984 to make Grenoble the site for the European Synchrotron Radiation Facility (ESRF) and the decision by the French and German governments in October 1984 to adopt this proposal, the first steps in the foundation of the new institute were taken at Grenoble during 1985. France, the Federal Republic of Germany, Italy and the United Kingdom have given a firm commitment to finance the new project, and it is hoped that some other countries will join them. Thus my predecessor's dreams are becoming reality.

Efforts are also being made towards a broader membership of ILL itself. A loose association with other countries in the coming year could be a first step towards a situation which may result in an ILL with more than three member countries after 1992.

## POSTSCRIPT

It seems that the first Annual Report of the new Director is also his last. He has followed the call of the new neighbour, the ESRF. In conclusion it only remains for me to thank all members of the ILL staff for a year which I have enjoyed from the first day to the last.

R. Haensel

# Rapport du Directeur

**B**ien que le Réacteur ait été à l'arrêt pendant une majeure partie de l'année et que le fonctionnement de routine n'ait repris qu'à partir du 3 septembre 1985, ce ne fut pas une année ennuyeuse, mais au contraire très active du premier au dernier jour.

## Programme de modernisation

Le programme de modernisation 1979-85 (deuxième souffle) est arrivé officiellement à échéance le 31 décembre 1985. Au cours des 11 mois d'arrêt du réacteur (3.10.84 - 2.9.85) des travaux complexes ont été menés à bien sur le réacteur même, dans le cadre du programme de modernisation. Avec l'échange des canaux H1/H2 et du canal transversal H6/H7, tous les éléments à l'intérieur du réacteur ont été renouvelés. Le collecteur de reprise, endommagé, a été extrait au moyen d'un procédé d'électro-érosion très sophistiqué, travaillant en immersion. La source froide verticale a été remplacée et la nouvelle version donne un flux de neutrons froids supérieur de 60 % à celui de l'ancienne source. Tous ces travaux difficiles ont été planifiés et exécutés par le Département Réacteur et se sont poursuivis sans incident en respectant parfaitement le planning établi.

Dans le cadre du programme de modernisation certains instruments ont été transformés, complétés ou améliorés ; ces travaux sont terminés pour la plupart et quelques instruments sont encore en cours de test.

Dans le hall réacteur les instruments D2 et D8 ont été remplacés par trois nouveaux instruments: D2B, D19 et D20. D9 a été rapproché de 3,5 m du coeur du réacteur. Grâce à une modification du monochromateur le flux d'IN8 a été amélioré d'un facteur 1,5. Le nouvel instrument IN20 remplace IN2.

Dans le hall des guides de neutrons l'appareil de diffusion aux petits angles D11 a été modifié. La mobilité du multidétecteur sous vide permettra un changement très rapide des paramètres de l'instrument et par conséquent une réduction importante du temps nécessaire pour passer d'une expérience à une autre. Les positions des instruments D1A et D1B sur le canal ont été inversées. Le guide entre D7 et IN10 a été modifié pour permettre l'installation du diffractomètre DB21 construit en collaboration avec le Laboratoire Européen de Biologie Moléculaire. Les choppers d'IN5 ont été modernisés et, après quelques difficultés dans la phase d'essai, fonctionnent maintenant de façon tout à fait satisfaisante.

Le projet le plus important du programme de modernisation, financé dans ce cadre, la deuxième source froide (horizontale), sera terminé en 1986, conformément au planning. Elle ne pourra vraisemblablement être mise en place qu'en 1987 car les instruments pour la deuxième source froide ont dû être retardés en raison des nombreux travaux sur et autour du réacteur pendant le grand arrêt.

Pour la deuxième source froide, un hall de guides a été construit pour abriter les instruments suivants : IN10C (version améliorée du spectromètre à rétrodiffusion IN10B); IN15, nouvel instrument d'écho de spin, qui sera construit en collaboration ILL, BMFT, KFA Jülich et HMI Berlin ; un autre instrument, D22, doit permettre d'alléger la charge très importante sur les appareils de diffusion aux petits angles. Un quatrième instrument sur la seconde source froide, IN14, spectromètre à trois axes, sera installé sur un guide de neutrons mais dans le Hall Experimental du Réacteur. Un cinquième projet pour la deuxième source froide doit être celui des Universités de Padoue-Pavie et Heidelberg : une expérience d'oscillation neutron-antineutron.

## Programme scientifique

Les améliorations qui ont suivi le programme de modernisation des instruments peuvent être décrites en trois grandes catégories. D'abord, un groupe d'instruments qui, lorsqu'ils seront tout à fait opérationnels, n'auront aucun équivalent à ce jour. A titre d'exemple, le nouveau spectromètre à écho de spin (IN15) est conçu pour avoir une résolution d'énergie de 0.1 neV et une valeur minimum de transfert de moment Q de  $10^{-3} \text{ \AA}^{-1}$ . Des études de la dynamique des solutions polymères, particulièrement à la frontière des diverses régions de diffusion, vont maintenant être possibles. IN15 peut être utilisé pour

étudier la dynamique critique au voisinage des transitions de phase magnétiques et structurales et pour l'investigation de la dynamique et de la relaxation de spin dans les systèmes de spins désordonnés et de faible dimension. Un autre instrument nouveau est le diffractomètre aux petits angles, DB21, pour la cristallographie des protéines. Les macromolécules et matériaux biologiques se présentent souvent sous forme de complexes de deux ou plusieurs espèces chimiques distinctes (les chromosomes, par exemple, consistent en protéine et ADN). La variation de contraste avec des mélanges  $\text{H}_2\text{O}/\text{D}_2\text{O}$ , utilisant la diffraction neutronique, est une méthode très efficace pour distinguer les divers composants à l'intérieur d'une macromolécule. Les méthodes classiques nécessiteraient une très haute résolution, ce qui, dans de nombreux cas, est une entreprise énorme ou même techniquement impossible. DB21 a été optimisé pour les études à faible résolution comportant le marquage H/D et les informations recueillies compléteront les études aux rayons X à haute résolution ou constitueront une contribution significative en elle-même.

Pour de nombreux groupes d'instruments tels que: spectromètres à trois axes, appareils à diffraction ou temps de vol, les performances ont été améliorées d'un ordre de grandeur. Sur D4B, le diffractomètre pour l'étude des liquides et des matériaux amorphes, les expériences de substitution isotopique peuvent être réalisées sur des systèmes très dilués: par exemple, le complexe de coordination formé par Cu dans  $\text{As}_2\text{Se}_3$  amorphe a été étudié à une concentration très faible (3 %at Cu). Plusieurs expériences ont été réalisées et des expériences supplémentaires proposées concernant les quasi-cristaux, c'est-à-dire ceux qui contiennent des phases icosaédriques et qui sont couramment constitués par la solidification rapide de certains alliages d'aluminium. Sur D1B, par exemple, une expérience récente a exploité la longueur de diffusion négative du manganèse et a permis, par variations de contraste (le fer étant substitué au manganèse) de recueillir directement des informations sur les corrélations Al-Al. Il est prévu maintenant d'exploiter les excellentes possibilités de D4B pour étudier cet alliage ainsi que les systèmes similaires susceptibles de former une phase quasi-cristalline par trempe rapide à partir des liquides. On peut ainsi espérer atteindre une interprétation microscopique de la stabilité de la phase icosaédrique. La nouvelle version d'IN2 (IN20), avec son très haut flux, permet de réaliser des expériences de polarisation inélastique. Des problèmes aussi différents que la dégénérescence apparente de certaines excitations magnétiques et de réseau dans  $\text{USb}$  et l'identification de magnons dans  $\text{RbFeCl}_3$  sont prêts à être abordés sur IN20. Les améliorations sur IN1 (spectromètre à trois axes sur la source chaude) permettent d'effectuer une expérience importante sur le nickel. Les modes d'onde de spin ont été étudiés lorsqu'ils rencontraient la région dense des excitations de Stoner. Il a été montré pour la première fois que l'onde de spin continue à se propager; un résultat d'une haute signification pour les théoriciens qui étudient les problèmes fondamentaux du ferromagnétisme dans le nickel. Ces expériences sont possibles car IN1 permet d'étudier des excitations à haute énergie à des valeurs relativement faibles de Q.

Enfin, plusieurs instruments ont été améliorés, à un degré certes plus modeste, mais qui permettront néanmoins de réaliser une recherche très novatrice. Par exemple, la science réalisable sur l'instrument bien connu à rétro-diffusion, IN10, a toujours été limitée par le faible flux. Les nouveaux cristaux analyseurs et le flux accru de la nouvelle source froide ont conduit à une amélioration d'un facteur 2 environ. Ceci permettra, par exemple, d'étudier de façon plus fine la dynamique de l'hydrogène dans les métaux puisque l'on peut utiliser des concentrations d'hydrogène plus faibles, d'où moins d'interaction hydrogène-hydrogène. De même, l'effet isotopique qui se produit lorsque l'hydrogène est remplacé par du deutérium peut aussi être mieux caractérisé. L'instrument à diffusion aux petits angles (D11C remplaçant D11), également amélioré, dispose d'un important dispositif nouveau: la distance entre l'échantillon et le détecteur peut être modulée de 1,2 m à 36 m. Par conséquent, des régions de transfert de moment Q de l'ordre de  $10^{-3}$  à  $10^{-1} \text{ \AA}^{-1}$ , qui méritent une étude détaillée, peuvent être étudiées immédiatement sans perturbation de l'échantillon ou de son environnement.

Le programme de modernisation aussi conduit à des innovations dans le domaine de la physique nucléaire et de la physique fondamentale. L'innovation la plus marquante a été l'installation de la nouvelle source à neutrons ultra-froids au-dessus du réacteur. Cette nouvelle source produit plusieurs centaines de fois plus de neutrons ultra-froids que toutes les autres sources existant jusqu'à présent. Au cours de l'année 1986 une demi-douzaine d'instruments doit être installée sur cette source.

En physique fondamentale, les efforts se sont poursuivis pour parvenir enfin à saisir la taille des constantes de couplage faible. Avec le spectromètre à désintégration de neutrons PERKEO nous avons réussi à déterminer avec précision le comportement des constantes de couplage des diverses parties des interactions faibles. De plus un vaste programme a été mis en place pour définir enfin avec plus d'exactitude la durée de vie des neutrons libres. La durée de vie des neutrons procure la seule possibilité, jusqu'à présent, de déterminer exactement la force globale de l'interaction faible. Il en résulte toutes les autres sections efficaces faibles et durées de vie qui, autrement, ne seraient pas directement mesurables et qui jouent un rôle important en cosmologie, en astrophysique et en physique solaire. A ce propos, il est à noter que l'on est arrivé à stocker pendant de très longues périodes des neutrons dans une bouteille à parois liquides.

Le groupe fission a pu étendre ses recherches de systèmes fissiles à d'autres actinides tels que le californium et le neptunium. On en retire d'intéressantes comparaisons avec les résultats issus des mesures réalisées jusqu'à présent de la fission de l'uranium. Les travaux relatifs à ce que l'on appelle la fission froide des noyaux ont été poursuivis. Pour les recherches de structure des noyaux l'ILL dispose, avec les instruments GAMS et BILL, d'appareils qui offrent la meilleure résolution au monde tant pour les rayons X et Gamma que pour les électrons. Avec ces appareils ont été poursuivies les mesures pour vérifier le modèle interactif des bosons. On a réussi, notamment, à identifier les transitions de monopole électrique. Grâce à l'installation du nouvel instrument GAMS 4, la résolution pour les rayons gamma a encore pu être améliorée de plusieurs ordres de grandeur.

Le groupe Physique Nucléaire a aussi réalisé le développement d'un nouveau type de détecteur qui, pour la première fois, ont été installés sur le spectromètre à produits de fission Cosi Fan Tutte, et avec lesquels il est possible de mesurer des neutrons et autres particules sur une grande surface de détection avec simultanément une très haute résolution de position et d'énergie. Il est vraisemblable que ces nouveaux détecteurs vont jouer un très grand rôle au niveau de toute la physique neutronique aussi bien qu'en physique nucléaire et des particules.

## Situation financière

Les difficultés budgétaires pour la recherche rencontrées par quelques pays partenaires ne sont pas restées sans conséquences pour l'ILL, mais elles ont cependant été moins sévères que pour bon nombre de laboratoires nationaux.

Afin de mieux maîtriser l'évolution du budget de l'ILL, les Associés ont mis en place un groupe d'experts chargés de les conseiller en matière d'économies et de priorités dans le programme scientifique de l'ILL. Les experts ont délibéré sur deux enveloppes budgétaires possibles et ont indiqué les postes qui pouvaient supporter une réduction de dotation sans porter véritablement préjudice à l'Institut. Les Associés ont ensuite autorisé un budget pour 1986 qui, à notre grande satisfaction, se situe au-dessus de ce plancher. En raison des grandes difficultés à résorber les 17,5 cases du programme de modernisation, les experts ont conseillé de ne pas procéder à des réductions de postes supplémentaires au cours des prochaines années. Naturellement, après vingt ans d'existence et suite à un certain nombre de mesures, un Institut n'a pas forcément la structure d'un organisme qui serait créé aujourd'hui avec la même taille et la même mission. L'approche de la structure idéale ne peut se faire qu'à long terme, mais ce modèle de structure idéale est également soumis à des modifications à long terme.

## Questions de personnel

Au 1er Avril 1985, un changement important est intervenu au niveau de la Direction : B.E.F. FENDER a quitté l'Institut, après une activité de 5 ans, pour prendre les fonctions de vice-chancelier de l'Université de Keele (Grande Bretagne). Son successeur vient de Kiel (FRA). Le Directeur adjoint allemand, le Professeur H. RUPPERSBERG, a quitté son poste après deux ans et demi pour retourner à son université d'origine à Sarrebruck. Quatre mois plus tard, le 1er Août 1985, il a été remplacé par un nouveau Directeur adjoint britannique, le Professeur J.E. ENDERBY de l'Université de Bristol. A. MICHAUDON a assuré la continuité pendant cette période de transition.

Le 23 Mai 1985 à Jérusalem, P. NOZIERES a reçu le Prix Wolf 1985 pour la physique ; un autre prix a été décerné à F. MEZEI, membre du personnel de l'ILL jusqu'au 31.7.1984, nommé depuis professeur titulaire à l'Université technique de Berlin : il s'agit du prix Hewlett-Packard 1986 récompensant ses travaux fondamentaux pour le développement des méthodes d'écho de spin, méthode qu'il a développée avec succès à l'ILL. Ce prix lui sera remis lors de la journée de l'EPS (Société Européenne de Physique) à Stockholm fin mars 1986.

W. PRESS ayant accepté les fonctions de professeur titulaire à l'Université de Kiel, D. RICHTER, de la Kernforschungsanlage Jülich, lui a succédé au poste de Senior Scientist. De même, D. DUBBERS (Université de Heidelberg) a remplacé F. GÖNNENWEIN (Université de Tübingen) au poste de senior scientist dans le domaine de la physique nucléaire et des particules élémentaires.

Indépendamment des cas cités plus haut, l'ILL est également fier de l'ensemble de son personnel. Les succès nombreux et divers des travaux sur et autour du réacteur, sur les instruments, en informatique, dans le domaine administratif, démontrent l'intérêt et l'enthousiasme du personnel qui, après 13 ans de fonctionnement, est toujours attaché à 'son' Institut comme au premier jour. Cet attachement s'est d'ailleurs manifesté par quelques réactions aux mesures d'économies que la Direction a été contrainte de prendre fin 1985.

## Plans futurs

Après la proposition de l'ILL au printemps 1984 d'implanter à Grenoble le futur Laboratoire Européen de Rayonnement Synchrotron (ESRF) et après que les Gouvernements de France et de République Fédérale d'Allemagne aient fait leur cette proposition, en octobre 1984, les premières actions en vue de la fondation de ce nouvel institut ont eu lieu au cours de l'année 1985 : la France, la République Fédérale d'Allemagne, l'Italie et la Grande Bretagne se sont engagées formellement à financer ce projet et nous espérons accueillir d'autres pays. Les rêves de mon prédécesseur sont donc en voie de réalisation.

En ce qui concerne l'ILL même, on aspire également à l'accueil d'autres pays partenaires. Une association dans des conditions plus souples avec d'autres pays serait une première étape qui pourrait conduire après 1992 à un ILL comprenant plus de trois pays membres.

## POSTSCRIPTUM

Il semble que le premier rapport annuel du nouveau Directeur doive être aussi son dernier. En effet, il a répondu à l'appel du nouveau voisin, l'ESRF. Il me reste donc à remercier tout le personnel de l'ILL pour cette année que j'ai pleinement appréciée du premier au dernier jour.

R. Haensel

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# **COLLEGES**

# Einleitung

Die gesamte wissenschaftliche Tätigkeit des Instituts ist in Kollegien mit spezifischen wissenschaftlichen Interessen aufgeteilt. Zur Zeit sind folgende Kollegien am Institut tätig :

Kollegium 2 : Theorie.

Kollegium 3 : Grundlagen- und Kernphysik.

Kollegium 4 : Strukturelle und magnetische Anregungen.

Kollegium 5 : Kristallographische und magnetische Strukturen.

Kollegium 6 : Flüssigkeiten, Unordnungen und Fehlstellen in Substanzen.

Kollegium 8 : Biochemie.

Kollegium 9 : Chemie.

Jedes Kollegium entspricht einem Unterausschuss des Wissenschaftlichen Rates des ILL, der die Direktion in Fragen des wissenschaftlichen Programms berät. Die Kollegien prüfen als

erste dem ILL unterbreitete Vorschläge zu neuen Forschungsvorhaben im Hinblick auf ihre technische Realisierbarkeit. Daraufhin werden sie den Unterausschüssen vorgelegt. Eine weitere Aufgabe der Kollegien ist es, zu einem reibungslosen Ablauf der Experimente beizutragen : Sie stellen den Gastforschern wissenschaftliche Hilfe vor Ort und beraten den Verantwortlichen der Experimentiereinrichtung und den Wissenschaftlichen Sekretär über die Zeiteinteilung für die Experimente an den verschiedenen Instrumenten. Die Sekretäre der Kollegien werden normalerweise für 2 Jahre gewählt, und halten mit der Direktion und den "Senior Scientists" regelmässig Sitzungen ab, um den notwendigen und alle wissenschaftlichen Tätigkeiten des ILL betreffenden Informationsfluss zu gewährleisten.

# Introduction

All the internal scientific activity of the Institut is grouped into "colleges" with particular scientific interests.

The following is the current list of colleges:

College 2: Theory.

College 3: Fundamental and Nuclear Physics.

College 4: Structural and magnetic excitations.

College 5: Crystal and magnetic structures.

College 6: Liquids, disorders and defects in materials.

College 8: Biochemistry.

College 9: Chemistry.

Each College corresponds to a Subcommittee of the Scientific Council of the ILL, which advises the Directors on the scien-

tific programme. New research proposals submitted to the ILL will first be examined by the Colleges with respect to their technical feasibility and then be presented to the Council Subcommittees. A further task of the College is to contribute to a smooth carrying-out of the experiments by appointing local contacts and by advising the instrument responsible as well as the Scientific Secretary on the time schedules for the various instruments. The College Secretaries are normally elected for a 2 year term and have regular meetings with the Directors and Senior Scientists to ensure the flow of information on all matters concerning the scientific activity of the Institut.

# Introduction

L'ensemble de l'activité scientifique de l'Institut est réparti en collèges avec des intérêts scientifiques spécifiques.

La liste actuelle des collèges se compose comme suit :

Collège 2 : théorie.

Collège 3 : physique fondamentale et nucléaire.

Collège 4 : excitations structurelles et magnétiques.

Collège 5 : structures cristallographiques et magnétiques.

Collège 6 : liquides, désordres et défauts dans les matériaux.

Collège 8 : biochimie.

Collège 9 : chimie.

Chaque collège correspond à un sous-comité du Conseil Scientifique de l'ILL, qui conseille la Direction en matière de programmes scientifiques. Les collèges examinent d'abord la

faisabilité technique des propositions de recherche soumises à l'ILL. Elles sont ensuite présentées aux sous-comités du Conseil Scientifique. Les collèges contribuent également au bon déroulement des expériences en proposant des collaborateurs locaux aux chercheurs invités, et en conseillant le responsable d'instrument ainsi que le Secrétariat Scientifique de la répartition du temps d'expérience sur les différents instruments. Les Secrétaires de Collège sont normalement élus pour 2 ans et se réunissent régulièrement avec la Direction et les "Senior Scientists" dans le but d'assurer une bonne circulation de l'information dans tous les domaines d'activité scientifique de l'Institut.

# COLLEGE 2 THEORY

## Members

T. Burkhardt  
H. Capellmann  
U. Felderhof  
D.R. Grempel  
R. Hasse  
F. Hirschfeld  
R. Jones  
P. Nozières  
J.M. Richard  
W. Schirmacher  
T. Sluckin  
J. Sölyom  
U. Stroth  
M. Uwaha  
T. Ziman

## Medium Term Visitors

E. Aker (RFA)  
F. Delyon (F)  
E. Eisenriegler (RFA)  
K. Fischer (RFA)  
S. Fishman (Israel)  
B. Gyorffy (G.B.)  
H. Hanley (U.S.A.)  
J.P. Hansen (F)  
B. Horowitz (Israel)  
G. Kendrick (G.B.)  
I. McDonald (G.B.)  
A. Megann (G.B.)  
H. Schulz (F)  
P. Schukla (India)  
S. Zouzou (F)

## INTRODUCTION

The scientific activity of College 2 covers a wide spectrum of interests in theoretical physics ranging from solid state physics through statistical mechanics to high energy physics. During 1985, in answer to a request from experimental groups, a special effort was made in the field of the physics of fluids, where a number of theoreticians carried out investigations. We have also received numerous visitors whose interests overlap with most of the subjects studied at the ILL. Continuing with the tradition, two members of the college, first Grempel and then Sölyom, were in charge of the organization of the general ILL colloquia. Grempel is now in charge of the weekly Theory Seminar organized jointly by ILL and CNRS. This seminar is the meeting place for the whole Grenoble community of theoreticians. Nozières gave a series of lectures on crystal morphology attended by numerous theoreticians and experimentalists from several Grenoble laboratories, and two series of lectures at the Collège de France, on kinetics of crystal growth and on superfluidity.

In April the Israeli parliament presented Nozières with the Wolf Prize for Physics 1985, rewarding him for his important work in condensed matter physics. Those of us having the opportunity of interacting with him on a day to day basis recognize how much this award is warranted.

## SCIENTIFIC ACTIVITY

### 1. Surface physics

Nozières and Uwaha continued with their work on crystallization of  $^4\text{He}$  and related problems. They have investigated crystallization waves, in particular in what way they are affected by superfluid flow parallel to the interface that causes the appearance of instabilities. They have pointed out several interesting experimental consequences of these instabilities and ways to control it. Nozières and Uwaha have also studied the mobility of kinks and steps at low temperature, the process

that determines that growth rate of vicinal faces, and problems related to sound transmission by the interface near the melting point and near the roughening transition.

Grempel has worked on the theory of atom-surface scattering by high index surfaces of solids. The calculation and measurement of the line shape and intensities of diffracted atom beams by a rough crystal surface, done in collaboration with an experimental group at Saclay, shows that the roughening transition may be recognized by anomalous behaviour of the temperature dependence of the Debye-Waller factor and singular behaviour of the line shape near a Bragg peak.

### 2. Disordered and stochastic systems

Grempel has worked on the effect of spin-orbit disorder scattering on the transport properties of dirty two-dimensional films, in particular temperature dependence of the conductance and of the magneto-resistance. Experimental consequences of this work are a reversal of the sign of the magneto-resistance at low fields and low conductance and inhomogeneous broadening of the conduction electron spin resonance line shape.

Felderhof has worked on a general cluster-expansion formalism for the electrical conductivity in a random medium, which is used to calculate the average resistance of a sample as well as its variance.

Grempel has continued his work on the transition to chaos in a certain type of driven non-linear oscillators and the crossover régime between classical and quantum mechanical behaviour. He has also been involved in the study of the statistics of energy level distributions in quantum systems, and the relationship between these statistical properties and the transition from regular to stochastic behaviour. In collaboration with H. Schuster he has investigated the effect of coupling to a phonon bath on the behaviour of pulsed non-linear oscillators. Summer stagiaire A. Magann started an investigation of the gap distribution in incommensurate metallic structures with the goal of calculating their low frequency optical absorption.

### 3. Magnetism

Several members of our group are actively involved in this field. Nozières and Schirmacher are studying the rôle of spin fluctuations in metallic systems with a special interest in almost ferromagnetic substances. They have found that there exists a characteristic temperature or, equivalently, a characteristic wave vector, at which spin fluctuations change their nature with important effects on the temperature dependence of thermodynamic quantities such as the specific heat and the magnetic susceptibility. This is especially important in view of the present interest in compounds such as  $\text{UPt}_3$  and  $\text{UBe}_{13}$  where spin fluctuations are believed to be important.

Capellmann is involved in work on iron and nickel in the paramagnetic phase in close collaboration with experimental groups at ILL. This research is centered on the experimental tests of the basic assumptions on which the fluctuating-band theory of itinerant magnetism is founded. In collaboration with Ziebeck, Neumann and others at ILL, Capellmann is working on the magnetic fluctuations in heavy fermion and intermediate valence materials. With his students, Thomz and Edmunds, he has investigated the possibility of coexistence of

triplet superconductivity and magnetic ordering and the way in which the two phenomena influence each other.

Sòlyom and Ziman continued their studies of the ground and low excited states of one-dimensional anisotropic quantum spin chains. Using variational methods and finite size scaling techniques they are trying to understand the phase diagram of these axially symmetric models, which describe the physics of compounds of the  $\text{CsNiF}_3$  family.

#### 4. Phase transitions

Burkhardt has been concerned with phase transitions and critical phenomena, in particular with boundary properties of systems close to a critical point. Using the newly developed theory of conformal invariance he has determined the order-parameter profile at the critical point induced by boundary fields for a variety of geometrical configurations. He has also studied the surface critical behaviour of magnetic systems. Sòlyom, in collaboration with B. Horowitz, has studied the effect of electron-electron interactions on the charge-density-wave formation in the Peierls transition as well as the modifications that these interactions produce on the phonon frequency and the electronic gap. He has also done work on phase transitions in generalized Ising models that include multispin interactions.

Sluckin has done work on the nematic to isotropic transition in liquid crystal films and on the switching transition in liquid crystal mixtures. With Ziman he has been involved in simulations of quantum spin systems that show similar critical behaviour.

#### 5. Physics of fluids

Due to the presence of a large number of specialists, this has been a very active field during 1985. Felderhof and Jones worked on the dielectric properties of suspensions of metallic particles in glass. Their results for the broadening and shifting of the plasmon resonance should be important for the interpretation of experimental results because they predict large deviations from the Clausius-Mossotti law. Felderhof and Matenn have worked on the theory of diffusion controlled reactions with the aim of obtaining a density expansion for the rate constant.

Jones has studied the problem of self-diffusion in colloidal suspensions and showed how dynamic light scattering can be used as a probe for phenomena on the length scale of the colloidal particles. Sluckin has been interested in the technologically important problem of the surface properties of charged colloids.

Schirmacher has done research on the thermodynamic properties of liquid metal-salt solutions with the aim of understanding the relation between the metal-nonmetal and phase separation transitions in these systems, as well as the asymmetries that show up in their phase diagrams. He has also worked on diffusion in glasses, resolving the long-standing problem of disagreement between the determination of activation energies by NMR and transport measurements.

Sluckin has been involved in a number of theoretical and experimental studies of liquid crystals and liquid crystal surfaces and interfaces. He has also worked on the calculation of the neutron scattering structure factor in mixtures of anisotropic molecules near an orientational order transition. Nozières and Quemada have continued with their work on the stability of flow patterns in the presence of particles in suspension.

#### 6. Nuclear and high energy physics

Hasse extended his work on semi-classical methods in nuclear physics to the calculation of effective masses, mean free paths and level densities. His results have been used in the interpretation of experimental results on single-particle depths. He has used the collisionally damped kinetic Landau equations to set up a theory of the width of giant nuclear resonances produced by two-particle, two-hole excitations.

Stroth and Hasse, in collaboration with P. Schuck, have analyzed experimental data of inelastic electron and proton scattering to extract the longitudinal and transverse response functions. Stroth has calculated the response functions taking into account the effect of collective excitations and particle-hole interactions. This work has established the importance of collectivity at high momentum transfer in the interpretation of  $(e,e')$  experimental data.

Richard has investigated various phenomena related to the interactions between antiprotons and nuclei with application to antiprotonic atoms and to antiproton-nucleus scattering. He has also worked on the calculation of baryon binding energies as functions of quark masses for flavour independent potentials. In collaboration with physicists of the ISN he has searched for possible multiquark states which are stable with respect to disintegration into mesons.

Secretary: D.R. Grempel

# COLLEGE 3 NUCLEAR AND FUNDAMENTAL PHYSICS

## Members of the College

### I. Internal Members

J.P. Bocquet  
H.G. Börner  
F. Blönnigen  
G. Colvin  
D. Dubbers  
H.R. Faust  
I. Förster  
P. Geltenbort  
F. Gönnewein  
H. Hanewinkel  
F. Hoyler  
C. Jewell  
W. Mampe  
G. Martinez  
P. Miranda  
J. Pannicke  
S. Robinson  
K. Schreckenbach  
U. Stöhlker  
H. Weikard

### II. Long-term Visitors

B. Alefeld (Jülich, KFA)  
M. Arnold (Heidelberg)  
J. Baumann (Bayreuth)  
R. Brissot (Grenoble)  
A. Bussière (Annecy)  
P. De Saintignon (ISN)  
W. El-Samman (Grenoble)  
S. Freedman (ANL)  
P. Grivot (ISN)  
I.A. Kilvington (RAL)  
E. Krüger (PTB Braunschweig)  
J. Last (Heidelberg)  
P. Liaud (Chambery)  
W. Nistler (PTB Braunschweig)  
M. Pendlebury (Sussex)  
H. Prosper (RAL)  
A. Steyerl (TU München)  
W. Weirauch  
(PTB Braunschweig)  
D. White (Monmouth Oregon)

## Collaboration of College 3 with Institutes and Universities in 1985

Algiers	Jülich KFA
Annecy	Karlsruhe KFK
Argonne	Karlsruhe University
Bayreuth	Köln
Belgrade	Leningrad
Bordeaux	Livermore LL
Braunschweig PTB	Los Alamos
Braunschweig University	Mainz
Brookhaven Nat.	Manchester
Lab. Bruyère-le-Châtel	Mol
Darmstadt GSI	Moscow Kurchatov
Darmstadt TH	Moscow ITEP
Fribourg	München TU
Geel JRC	München University
Giessen	Rutherford Laboratory
Grenoble CEN	Seattle
Grenoble ISN	Sofia
Grenoble University	Surrey
HarvardHarwell	Sussex
Heidelberg	Tübingen
Imperial College London	Washington NBS

## GENERAL SUMMARY

During 1985, the main scientific effort was directed towards the analysis and interpretation of experiments performed

before the reactor shut-down. After the successful start on schedule of the reactor at the beginning of September, experimental activities on all facilities began immediately. The main fields studied by College 3 are outlined below:

1. **Thermal neutron induced fission** was studied at the parabola spectrometer Lohengrin (PN1) and at the energy-time-of-flight spectrometer COSI FAN TUTTE (PN8).

2. **Nuclear structure studies** have been performed by the electron spectrometer BILL (PN2), the curved crystal spectrometers GAMS 1, 2/3 (PN3), the pair spectrometer PN4 and the on-line mass separator OSTIS (SN6).

3. In the **Fundamental Physics** domain a completely new and very powerful ultra cold neutron-very cold neutron (UCN-VCN) facility (see instrumental part of this report) has been added to the existing SN5 source and the polarized beam facility SN7.

## SCIENTIFIC TRENDS AND HIGHLIGHTS IN 1985

### Fission

The evaluation of experiments in thermal neutron induced fission performed in 1984 at the fission fragment spectrometer COSI FAN TUTTE (PN8) has progressed well and is still underway. The first results of these experiments were presented at the Santa Fe conference on 'Nuclear Data for Basic and Applied Science'.

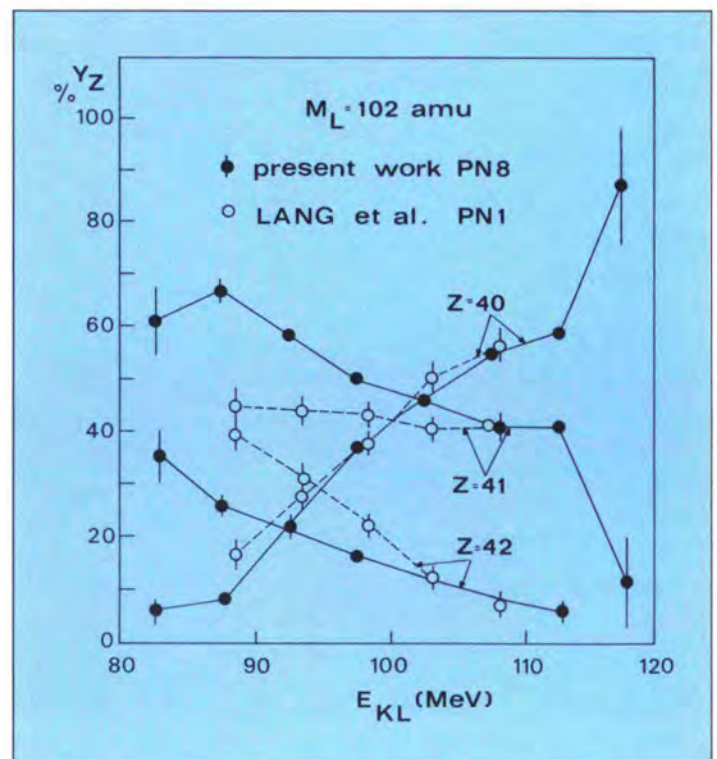


Figure 1: Relative nuclear charge yield for a fixed light fragment mass ( $M_L = 102$  amu) as a function of its kinetic energy  $E_{KL}$  in the thermal neutron induced fission of  $^{235}\text{U}$ . For cold fission ( $E_{KL} > 112$  MeV) the fragmentation of the compound nucleus  $^{236}\text{U}_{144}$  into the fragment-pair  $^{102}\text{Zr}_{62}/^{134}\text{Te}_{82}$  dominates.

A notable result has been obtained by a Bordeaux-ILL collaboration, showing for the first time that in the limiting case of cold fragmentation, single nuclear charges are selected by the fission process (see figure 1). As a consequence, theoretical models predicting a non vanishing variance of the nuclear charge distribution based on zero point oscillations have to be put into question.

For the more exotic  $^{245}\text{Cm}(n_{th},f)$  reaction, mass and energy distributions of fragments as well as kinetic energy and angular distribution of coincident Long Range Alpha particles (LRA) have been measured. The mean angle of the LRA's with respect to the light fragment path was found to be 84.2 degrees, while their most probable energy is 16.6 MeV. The mass yields of light fission fragments agree fairly well in the range between 80 and 100 amu with those obtained by radiochemical methods, while around mass 110 the present values are up to 40% higher.

A considerable part of the beam time on LOHENGRIN (PN1) was used to complete the studies of  $^{237}\text{Np}(2n_{th},f)$  fission with data on cold fragmentation and on nuclear charge distributions at different kinetic energies. These measurements constitute the first data set of a fissioning odd proton system including information on mass, kinetic energy, nuclear charge and ionic charge for the group of the light fission fragments. As was expected the nuclear charge distribution of  $^{237}\text{Np}(2n_{th},f)$  fission follows a smooth trend and shows no odd-even effect, whilst in the thermal neutron induced fission of even proton systems up to Pu this effect is clearly marked (see figure 2). With the large ionization chamber of PN1, measurements of masses and kinetic energies have been pushed into the low yield symmetric region in  $^{239}\text{Pu}(n_{th},f)$  fission. The data aim at shedding light upon the mechanism of the symmetric fission mode which looks very different from the asymmetric mode in terms of excitation functions and measurable quantities of the fission fragments.

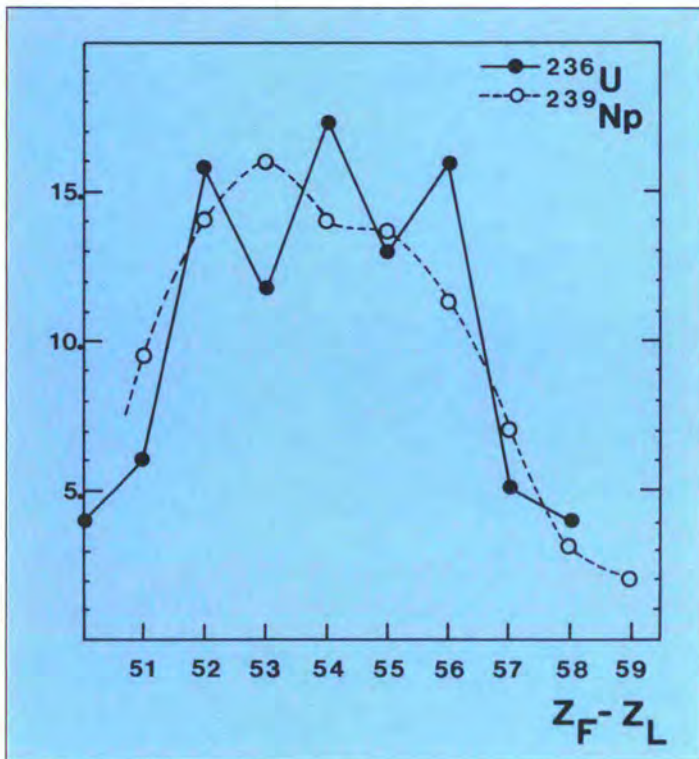


Figure 2: Preliminary nuclear charge distribution of  $^{237}\text{Np}(2n_{th},f)$  measured at the most probable kinetic energy and compared to the nuclear charge distribution of  $^{235}\text{U}(n_{th},f)$ .

The studies of the slowing down process of heavy ions in gases have been continued on Lohengrin using a high resolution axial ionization chamber of the type installed at PN8. In this type of chamber the incoming ions produce an ionization track along the electric field lines, the so-called Bragg curve. Therefore the signal pulse shape at the grid-screened anode is just a mirror image of the Bragg curve. Figure 3 shows individual Bragg curves obtained by digitizing the anode signal event-by-event. The large fluctuations of the ionization density were traced back to atomic collisions. They impose an upper limit to the charge resolving power  $\Delta Z/Z$  which can be achieved by means of such an axial ionization chamber. By optimizing the ratio between track length and grid-anode distance, a resolving power  $\Delta Z/Z = 46$  for  $Z = 39$  of fragments with  $A = 97$  amu and  $E = 100$  MeV was measured.

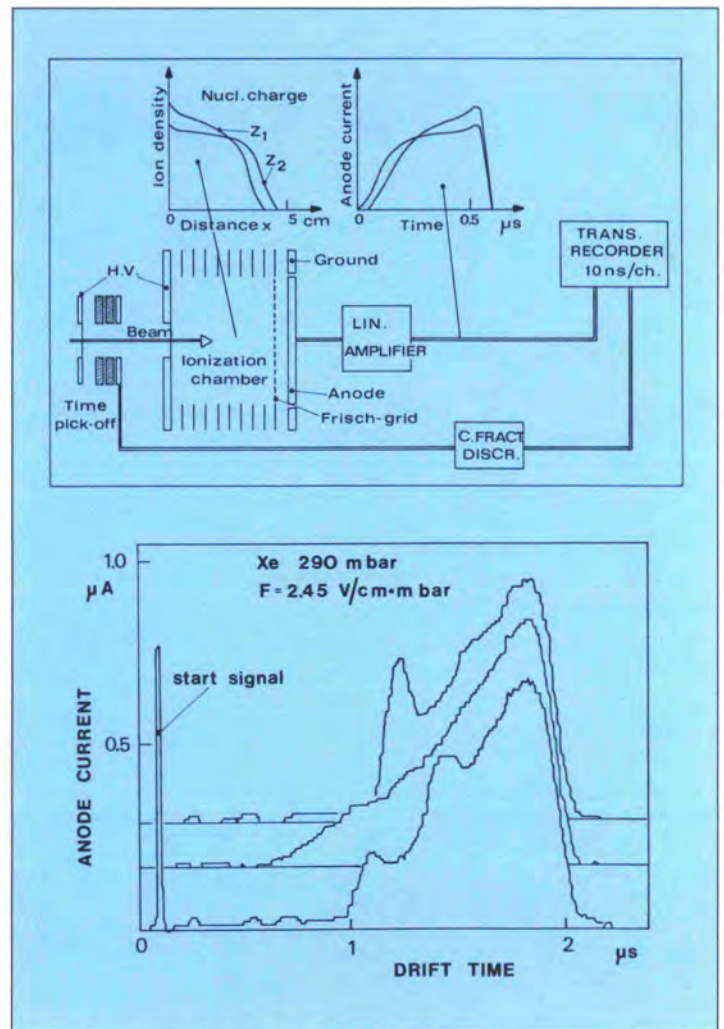


Figure 3 a): Velocity selected fission fragments are stopped in an axial ionization chamber. The anode signal which corresponds to the Bragg curve is digitally stored. A trigger signal is delivered by an external time pick-off.  
b): Bragg curves of 3 heavy fission fragments with the same mass number  $A = 139$  amu and the same kinetic energy  $E = 68$  MeV. Xe (with 10% methane) was used as counter gas.

## Nuclear structure

Work performed in this field can be roughly grouped into:  
i) measurements on the properties of nuclear states with the aim of testing the Interacting Boson Approximation (IBA), Interacting Boson Fermion Approximation (IBFA) or other models

ii) measurements of electric monopole (E0) transitions  
 iii) construction of 'complete' level schemes and  
 iv) spectroscopy of nuclei far from stability  
 Although double neutron capture has been discussed previously in the ILL Annual Reports, it is still worth noting that this technique is only possible where high neutron fluxes are available. In this respect BILL, GAMS and PN4 have to be considered as unique instruments.

The Pt-Ir-Os nuclei have been extensively studied in recent years in order to confirm (or to refute) predictions arising from the IBA (or IBFA) in the  $O(6)$  symmetry limit of this model. It appeared from earlier results that with increasing neutron number from  $^{191}\text{Ir}$ , the spin (6) symmetry of the IBFA was progressively broken. The recent results from  $^{193}\text{Ir}(2n,\gamma)^{195}\text{Ir}$  at least show  $^{195}\text{Ir}$  to be a good example of the spin (6) scheme and is now in fact considered to be the isotope possessing the most complete  $\sigma < \sigma_{\text{max}}$  level scheme yet measured (see figure 4). The identification of double neutron capture lines from the many  $^{194}\text{Ir}$  single capture lines (n-capture cross section of  $^{193}\text{Ir} \sim 100 \text{ b!}$ ) was a difficult task. The BILL spectrometer was probably the most useful instrument for this purpose, as direct subtraction of time varying spectra was possible, thus clearly indicating the 'growing-in' lines of  $^{195}\text{Ir}$ .

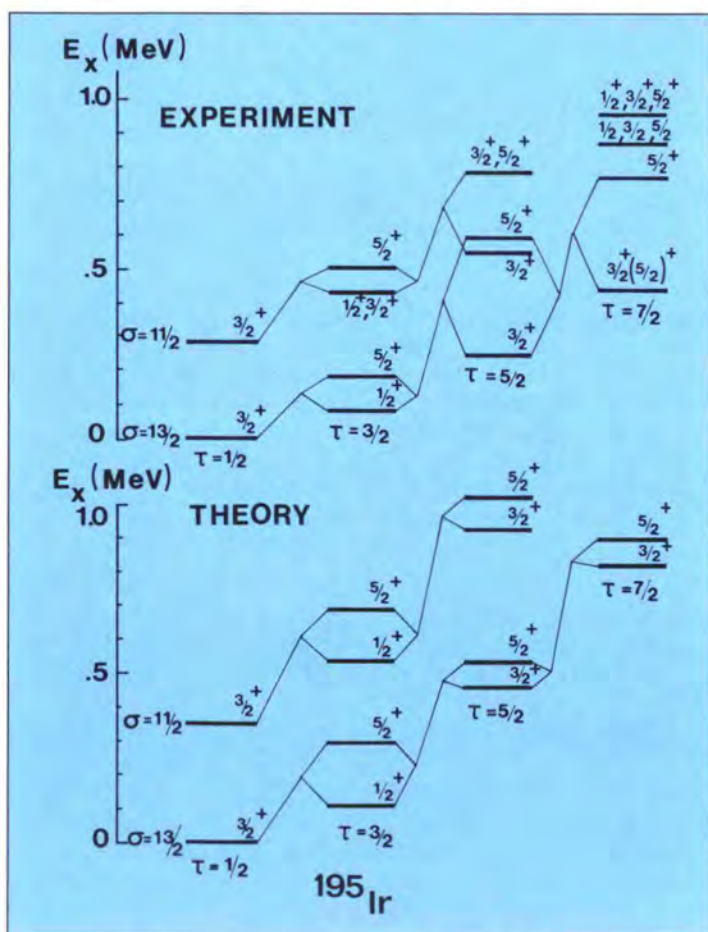


Figure 4: A comparison in  $^{195}\text{Ir}$  of both experimental and theoretical level energies. Spin (6) quantum number and  $J^\pi$  assignments clearly show a one to one correspondence, even for  $\sigma < \sigma_{\text{max}}$ .

Previously obtained experimental results from the  $^{171}\text{Yb}(n,\gamma)^{172}\text{Yb}$  reaction were compared with calculations made in the framework of IBA-1, especially with respect to low-lying positive parity levels. It was concluded that  $^{172}\text{Yb}$ ,

despite its location in the centre of the rare-earth region, if globally viewed, is not well described in a purely collective framework (collaboration with the University of Manchester). In a Munich-ILL collaboration primary capture  $\gamma$ -rays following multiple neutron capture in  $^{151}\text{Eu}$  have been measured at PN4 as a function of the irradiation time (figure 5). In this way, the neutron capture cross sections of the  $^{152}\text{Eu}$   $3^-$  ground state and of the  $^{152}\text{Eu}$   $0^-$  isomer were determined. These cross sections are relevant for the interpretation of inelastic scattering of neutrons at isomeric states with energy gain (neutron acceleration).

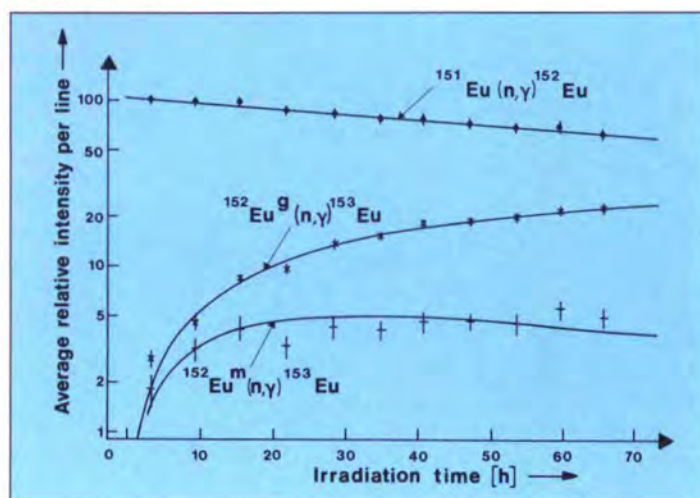


Figure 5: Average relative intensities of primary transitions of the reactions  $^{151}\text{Eu}(n,\gamma)^{152}\text{Eu}$ ;  $^{152}\text{Eu}^g(n,\gamma)^{153}\text{Eu}$  and  $^{152}\text{Eu}^m(n,\gamma)^{153}\text{Eu}$  as a function of the irradiation time.

The interpretation of measurements in the Dy-region performed on GAMS was continued, especially of the odd deformed nucleus  $^{161}\text{Dy}$ . The study of such nuclei provides an excellent opportunity to acquire an extensive set of data for investigating various modes of excitations in nuclei, such as single particle, rotational and vibrational excitations. A level scheme of  $^{161}\text{Dy}$  has been constructed which is believed to be essentially complete below 1 MeV concerning  $1/2^+$  and  $3/2^+$  levels. 13 rotational bands have been assigned.

The measurement of E0 transitions in nuclear spectroscopy has been a recurring theme for experiments proposed for the BILL spectrometer. High resolution electron spectroscopy is in fact one of the few ways available for detecting these transitions. For example, E0 transitions in  $^{124}\text{Te}$  have been carefully studied on BILL for a sensitive comparison between the IBA and DDM (dynamic deformation model) models, both of which have characteristic and often conflicting predictions for E0 transition strength. In this analysis, the DDM was shown to be more able to reproduce the  $0^+$  levels as  $K^\pi = 0^+$  members of a collective band without the need to invoke quasi-particle excitation. Both  $B(E2)$  and  $X(E0/E2)$  values were also satisfactorily reproduced. (Collaboration with Sussex). Recent E0 measurements have included  $^{106}\text{Pd}$ .

Within the framework of the systematic studies of odd-mass  $A \sim 100$  nuclei at OSTIS (SN6), the low-lying levels in  $^{99}\text{Y}$  have been investigated. From a comparison of the experimental results with the particle quadrupole phonon model (PTQM) and with RPA (Random Phase Approximation) shell model calculations using both Nilsson model and Woods-Saxon wave functions, the proton configuration  $[422\ 5/2]$  for the ground band was confirmed, and several one-quasi-particle states up to about 1 MeV in  $^{99}\text{Y}$  were located (figure 6). Among these,

the [303 5/2], [431 3/2] and [431 1/2] states were identified as band heads of excited rotational bands. The energies of the 1qp-states together with their  $\beta$ -feeding have induced further conclusions on the Nilsson parameters  $\nu$  and  $\mu$  in the  $A \sim 100$  mass region and the determination of the quadrupole deformation of  $^{99}\text{Y}$  to  $\epsilon_2 = 0.33 + 0.01$ . With a  $\log ft = 5.0$ , the  $^{99}\text{Sr}$  [411 3/2] ground state to  $^{99}\text{Y}$  [422 5/2] ground state  $\beta$ -transition is the fastest of the type 'allowed-hindered' observed so far.

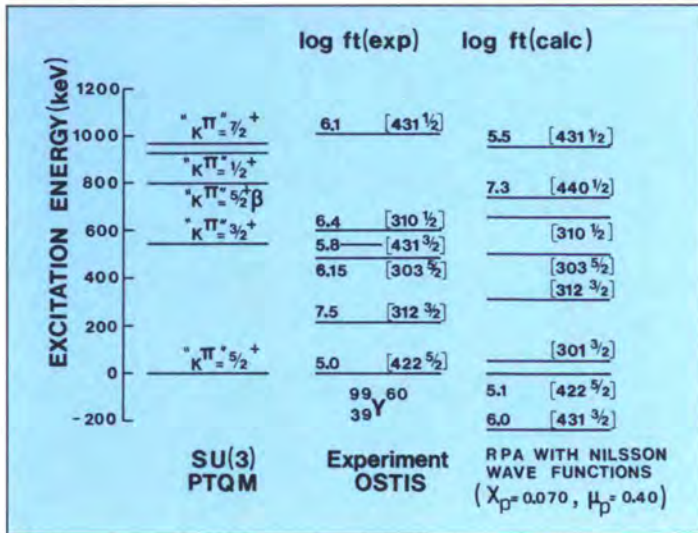


Figure 6: One-quasi-particle configurations in  $^{99}\text{Y}$

Using the high-temperature ion-source at OSTIS, the yields and isomeric ratios of neutron-rich In isotopes were determined. The isomer ratios show a remarkable oscillation between preferential feeding of high-spin states of odd-mass In-isotopes and low-spin states of even-mass nuclei (see figure 7). Whilst the values of the fraction of the odd-mass high-spin isomers agree surprisingly well with an older statistical model prediction, the corresponding values for the even-mass high-spin isomers appear to be considerably lower than the statistical estimates. These deviations from the expected behaviour may be due to a non-statistical de-excitation of the initially highly excited fission fragments. The same type of discrepancy has also been observed in the odd-odd isotopes,  $^{96}\text{Y}$  and  $^{98}\text{Y}$  at LOHENGRIN.

Furthermore, as a first on-line test of a recently developed negative ion source, a directional correlation measurement of  $^{86}\text{Br}$  was performed. As an encouraging result, five transitions of the  $\beta$ -daughter  $^{86}\text{Kr}$  have been observed. The spin assignment of the first two excited states were confirmed. This work is still in progress.

With the recently installed double flat crystal spectrometer GAMS 4, a new value for the deuteron binding energy of  $B(D) = 2.3881768(24) \cdot 10^{-3}$  amu has been determined based on an absolute wavelength determination of the 2.2 MeV n-p capture  $\gamma$ -ray (see box in this report). From this, a derived value for the neutron mass of  $M_n = 1.008664928(11)$  amu was obtained.

## Fundamental Physics

- Work has been concentrated in two main areas:
- improvement, development, installation and tests of UCN-VCN sources, and
  - determinations of the neutron life-time

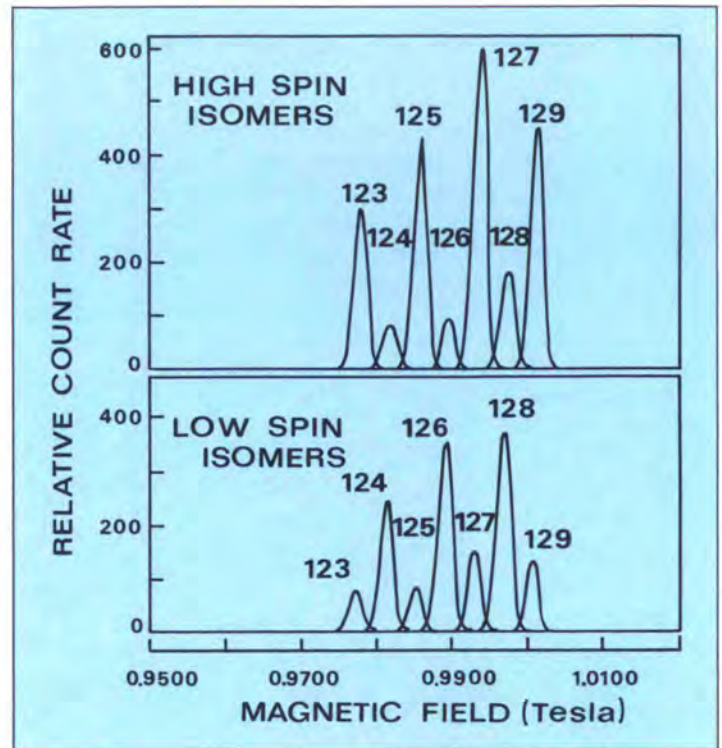


Figure 7: Mass spectrum of high- and low-spin isomers of the In-isotopes 123-129 corrected for different detection efficiencies (peak areas = relative fission yields)

The UCN-VCN source SN5, installed in 1976, has been very successful. The in-pile guide section however, exposed to a high radiation level and connected to an unsatisfactory vacuum system has degraded with time. Therefore in 1985 the available replacement source was commissioned and the pumping system improved. The UCN density as measured in a stainless steel bottle was  $0.6 \text{ cm}^{-3}$ , not corrected for detector efficiency and transmission losses. At the same time the EDM (electric dipole moment) experiment detected 6 times more neutrons than during its last run on the old source.

Although no further EDM data had been taken during 1985, the processing (at Sussex) of all previous runs performed at ILL improved the experimental limit for the neutron EDM to  $(-1.8 \pm 2.9) \cdot 10^{-25} \text{ e.cm}$ .

The EDM experiment, limited up to now by neutron counting statistics, has been improved in all other respects to accept a 200 times higher UCN density. This was a necessary step before moving the experiment to the much stronger UCN source T-TGV (Turbo Tube Guide - or Garching - Vertical). The limit there will probably be given by the magnetometer noise.

The liquid UCN He source has experienced improvements on the cryogenic and UCN extraction side as described in more detail in the instrumental section of this report. It will be transferred to the cold beam H17 to profit from the higher UCN density and to carry out further  $^4\text{He}$  research and development work.

The new vertical UCN-VCN source T-TGV has been installed at Niveau D of the reactor (for more details see instrumental part). UCN bottle measurements at the exit of the TGV feeding the turbine, carried out under identical conditions as on SN5, yielded a detected density of  $\sim 41 \text{ UCN} \cdot \text{cm}^{-3}$ . The gain factor due to the Garching turbine is still open. Detailed time of flight and gold foil activation measurements revealed a gain factor of about 75 compared to SN5, due to the new Cold

Source and the excellent guide. The maximum intensity is at about 60 Å. The out-of-pile part has been designed and constructed at Garching and has been financed mainly by the BMFT. This new facility will open up a new dimension in the UCN field and in the almost completely unexploited domain between 30 Å and 100 Å.

The first proposed experiments to be installed on platforms near the turbine are: the UCN microscope (Garching), the EDM experiment (Sussex, Harvard, University of Washington, RAL, ILL), the neutron storage ring (Bonn) and a test facility. The gravity spectrometer NESSIE and a VCN microscope (both from Garching) will follow. In spite of this heavy experimental load the UCN-VCN source will be made available to further experiments.

The installation of an improved neutron charge experiment (Bayreuth) on the cold beam H18 started in August 1985. The set-up makes use of achromatic neutron focussing mirrors and multi-slit systems. The design sensitivity is of the order of  $10^{-21}$  electron charges.

On the superconducting spectrometer PERKEO, installed at the SN7 position, the neutron decay programme has been continued. The instrument was running in 1985 with unpolarized neutrons for a direct determination of the neutron life-time. A systematic study of the different measuring methods possible with PERKEO was performed using alternatively chopped or continuous, white or monochromatic neutron beams, and different types of thin or thick neutron detectors, with the aim of finding out the technique giving the smallest systematic error.

The set-up of a second, completely different neutron life-time experiment has been continued at the test position T16 at the neutron beam H14. The apparatus consists of an in-beam  $^4\text{He}$  drift chamber ensuring a  $4\pi$  detection of decay electrons out of a through-passing neutron burst. A small admixture of  $^3\text{He}$  to the counter gas provides a neutron flux measurement in the same detector. The ratio of electrons from the neutron beta decay to  $^3\text{He}$  ( $n, p$ ) events determines directly the neutron life-time. The drift chamber operates similarly to a time projection chamber and thus recognizes the particle tracks. Only those tracks starting inside the neutron burst are accepted as a possible real event. Presently a double-chopper shaped as a drum and a new  $\text{RbC}_8$  monochromator are being installed. A further improvement of the radiation shielding and the drift chamber electronics is envisaged.

Another field of interest has been systems of 'dressed neutrons': when polarized neutrons are exposed to a large number of radio-frequency quanta, several peculiar effects may arise. For example the g-factor of the dressed neutron may be very different from the g-factor of the free neutron, which may even become zero or change sign. These effects were recently investigated in a neutron spin rotation experiment. It was shown that a static magnetic interaction of the neutron can be completely decoupled by the superposition of a radio-frequency field of appropriate strength, but with rather arbitrary frequency (figure 8).

In another study being performed at the electron spectrometer BILL (PN2), the source spectrum of reactor antineutrinos  $\bar{\nu}_e$  from  $^{235}\text{U}$  fission products was deduced from an accurate measurement of the correlated  $\beta$ -spectrum. The measurements were carried out at low (4 MW) and normal (57 MW) reactor power to optimize the signal to background ratio for the  $\beta$ -spectrum and the sensitivity for the intensity calibration, respectively. As shown in figure 9, the intensities were calibrat

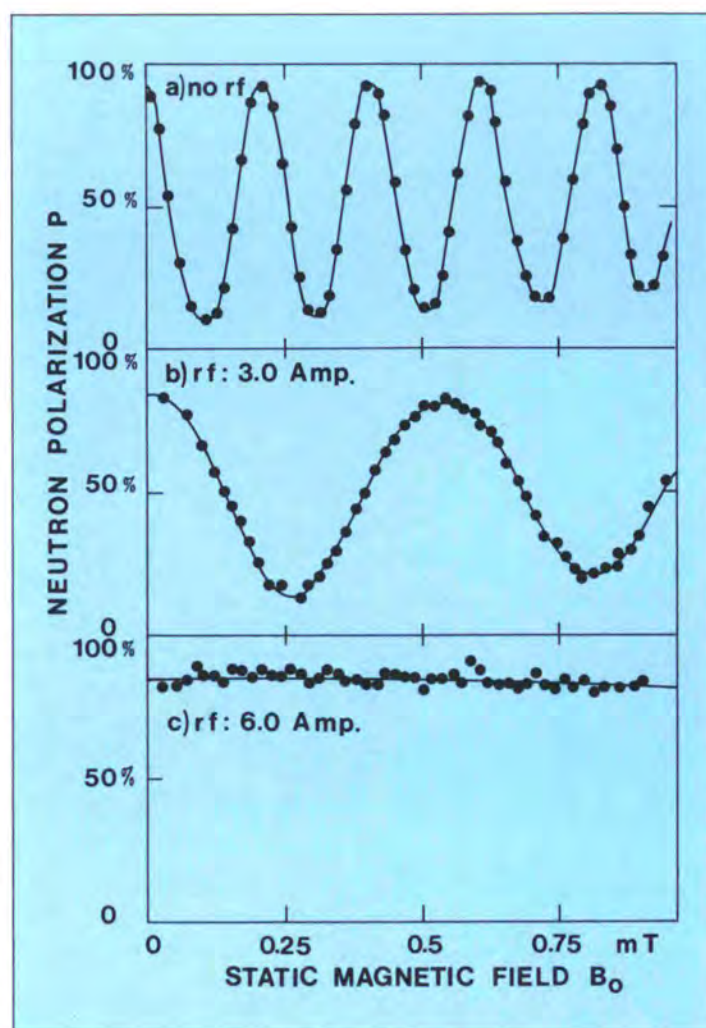


Figure 8: Spin rotation measurements with dressed neutrons: By radio-frequency (rf) irradiation, the spin-rotation process can be slowed down (b), and even be completely stopped (c).

ed over the whole energy range up to 9 MeV. The precision was improved to 3% for the absolute rates per fission. The knowledge of the  $\bar{\nu}_e$  source spectrum is of great importance for neutrino oscillation experiments recently carried out, which use nuclear reactors as an intense source of electron antineutrinos. This is true even for such cases where the search for neutrino oscillations was performed by  $\bar{\nu}_e$  detector measurements at several distances from the reactor core.

## Special beam experiments

The systematic study of the ( $n_{th}, \alpha$ ) and ( $n_{th}, p$ ) reactions has been continued at the H22D facility. New reliable results have been obtained for the  $^{50}\text{V} (n_{th}, p)^{50}\text{Ti}$  reaction which considerably improve previous data.

At the H22E beam position a test experiment of a new method in searching for PNC (parity non conserving) effects has been performed by a Kurchatov Institute-ITEP (Moscow)-ILL collaboration. The preliminary results are encouraging and it seems that resonance scattering of circularly polarized gammas will be a new tool to analyse PNC effects in nuclei.

The analysis of life-time measurements of nuclear levels excited via the ( $n, \gamma$ ) reaction has progressed (figure 10). As a

notable result, the half-life of the  $K^\pi = 0_3^+$  level at 1405 keV excitation energy in  $^{172}\text{Yb}$  was measured as  $t_{1/2} = 0.42$  (6) ns using the generalized centroid-shift method. The  $B(E2)$  value for the transition of this level to the first excited state in  $^{172}\text{Yb}$  is extremely low ( $3 \cdot 10^{-3}$  W.U.). Possibly this level can be interpreted as a mixed symmetry state as predicted by the IBA - 2 model.

The Instrumental Neutron Activation Facility continued its operation with irradiations carried out at the Melusine reactor. Studies for trace and rare element concentrations were undertaken on a series of rocks from the Andian Cordillera, Germany, Australia and Canada.

### Workshops

Two workshops were organised in 1985 at the ILL by College 3 in close cooperation with SCAPRO.

The first was held in January and was devoted to 'Future plans in nuclear spectroscopy at the ILL high flux reactor', organised by F. Hoyler. It was attended by some 30 participants from the ILL user community. The aim of the workshop was to inform the users of improvements and new possibilities on the different instruments and to provide a platform for fruitful interchanges of ideas about future projects in the broad field of nuclear spectroscopy.

The second workshop on 'Lectures on weak nuclear interaction' took place in October and was organised by B. Desplanques, P. Geltenbort and F. Gönnerwein. The aim of this series of lectures was primarily educational for the College 3 staff. For both workshops presented view graphs were published as internal ILL reports.

Finally, the annual meeting of the Nuclear Energy Agency Nuclear Data Committee of the OECD took place partly at the ILL. In a series of talks the instrumental possibilities and the scientific activities at the ILL in the field of nuclear fission, nuclear spectroscopy and fundamental physics were presented to this international committee by J.P. Bocquet, F. Hoyler and D. Dubbers.

### Seminars

As in the past, a series of seminars dealing with topics of specific and general interest to College 3 has been held.

Secretary: P. Geltenbort

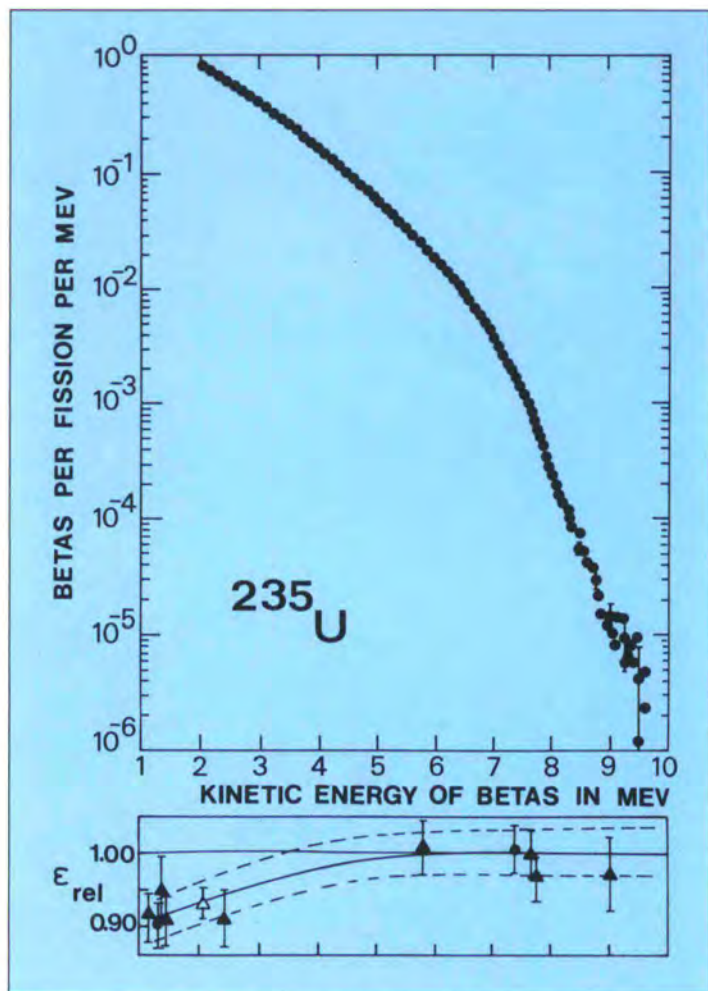


Figure 9: Experimental beta spectrum of  $^{235}\text{U}$  fission products. The error bars in the spectrum illustrate the statistical accuracy for bins of 50 keV (90% CL). In the lower part the determined efficiency curve  $\epsilon(E_\beta)$  with the uncertainty range (shaded area) is shown.

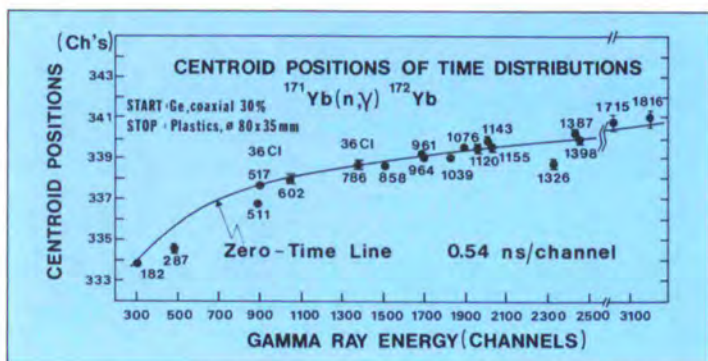


Figure 10: Centroid diagram demonstrating the half-life measurements in  $^{172}\text{Yb}$ . The 287 and 1326 keV transitions depopulate the 1405 keV  $0_3^+$  level having a life-time of 0.42 ns and deviate therefore from the zero line.

## The Scientific and Technological Spin-off from Free Neutron Experiments at ILL

At ILL a number of experiments are concerned with the particle properties of free neutrons. The measurements are performed with specially designed instruments that are part of the fundamental physics programme at ILL.

These experiments are interesting for several reasons: firstly, there is a general metrological interest to know nature's constants with high accuracy; secondly, some of the properties of the neutrons are of central importance in modern unified theories; thirdly, technological progress often results from scientists pushing the limits of experimental techniques, and there is an important impact from these experiments on other fields of science and technology. It is mainly this third point that will be discussed here.

The accompanying table lists some of the free neutron's properties which have been measured at ILL in recent years. The table is set up only for illustration, and is not meant to be the newest release of the neutron's fundamental properties. Most experiments were performed and financed by visiting groups, see also the last column of the table. Some of the values listed ( $d_n$ ,  $\tau$  direct, D, b) have also been measured at other reactors with similar accuracy.

The first property listed, the **neutron mass**, is determined from the masses of the proton and the deuteron. The main error comes from the uncertainty in the deuteron's binding energy. The new special instrument GAMS4 determines the wavelength of the gamma-ray emitted during deuteron formation in terms of the laser wavelength standard, thus establishing a direct experimental link between all frequencies from 1 to  $10^{21}$  Hertz. To accomplish this, the lattice spacing of silicon and other crystals were defined in terms of the laser wavelength, by simultaneous optical and neutron interferometry. Further, new standards in mechanical positioning were set by this instrument: the apparatus allows the optical measurement of rotations as small as  $10^{-9}$  rad, and the control of displacements as small as 0.01 Ångstrom.

The ratio of fundamental constants  $h/m = \lambda \cdot v$  is measured on S50. The wavelength  $\lambda$  and the velocity  $v$  of the neutrons are independently determined to high precision (aim:  $10^{-6}$ ) using several ingenious methods, which may become useful for the next generation of very high resolution neutron scattering instruments.

To set limits on the **charge of the neutron**, the lateral displacement of a neutron beam under the influence of an electric field is measured. This accomplishment is non-trivial, because a neutron beam is essentially a jet of diffuse gas. Nevertheless, a 10 Ångstrom displacement of this gas jet can be measured after a flight path of 10 meters with the new set-up, and therefore this instrument is, by itself, a major achievement of experimental science. It was necessary to develop a whole line of new and elaborate neutron optical equipment for this purpose.

The apparatus for the **electric dipole moment of the neutron** can measure neutron energy shifts as small as  $10^{-21}$ eV or  $10^{-6}$  Hertz. This achievement required, among others, the development of the ultimate standards in: ultra-cold neutron storage; atomic resonance magnetometry; magnetic shielding; and creep free high electric field generation. The sensitivity of the apparatus has steadily increased by one order of magnitude every seven years over the last 35 years, and there

is both scientific as well as technological incentive to continue for at least another 20 years. Also the development of high intensity ultra-cold neutron sources was in part motivated by this project.

The determination of the weak interaction coupling constants from **neutron beta-decay** is interesting not only for particle physics. The cross sections of many weak processes can only be calculated from the neutron lifetime. Thus, the uncertainty in the neutron lifetime today contributes the main error in calculations on the formation of matter in the early universe, and significant errors in calculations of the solar hydrogen burning cycle, the solar neutrino problem, and in some calculations on the evolution of stars. Neutron decay experiments are difficult, and as the demand on the precision of the weak coupling constants increases steadily, these experiments will continue to be with us also in the future. - Neutron decay experiments further have stimulated developments in detector technology, in absolute neutron flux determination, and in magnetic and fluid-wall neutron storage devices.

The first **neutron oscillation** experiment at ILL set a limit of  $\tau_{n\bar{n}} > 10^6$  sec to the oscillation period (while the time-of-flight of the neutrons was only  $10^{-2}$  sec). We expect to push this limit to  $10^8$  sec, corresponding to a coupling strength  $< 10^{-23}$  eV, which will require new developments in neutron guide technology (large divergent non-magnetic guide), large scale magnetic shielding (60 m<sup>3</sup> shielded to  $< 10$  Nano tesla), and precision particle tracking, and may also promote technology transfer between high energy and neutron laboratories.

**Neutron interferometry**, which has allowed for many high precision tests in fundamental physics, is in itself a big technological achievement; it also has established precise neutron scattering amplitude standards, some of which are listed in the table, and which are useful in neutron scattering work in general. Similar precision has been obtained with the Garching neutron gravity spectrometer, which will be installed at the new ultra cold neutron facility now being set up at ILL by the Garching group.

Further high precision experiments are concerned with **parity violation in the nucleon-nucleon interaction**. Thus, the rotation of the neutron spin in an isotropic medium can now be measured with  $10^{-6}$  rad accuracy, which will be increased to  $10^{-8}$  rad in future experiments.

In summary, the high precision measurements of the neutron's particle properties are not only of great importance in particle and fundamental physics, but they are also valuable for the advancement of other fields of science and for the development of new technologies, both in the field of neutrons and elsewhere.

**Table of recent measurements of neutron properties at ILL**

Property	Value	Year of publication	Collaboration
mass	$m = 1.008664928 (11) \text{ a.m.u.}$ from: $E_B (\text{deuteron}) = 2.3881768 (24) \cdot 10^{-3} \text{ a.m.u.}$ $h/m = \lambda v = 3.956093 (63) \cdot 10^{-7} \text{ m}^2 \text{ s}^{-1}$	1985 1984	NBS Washington-ILL PTB Braunschweig
charge	$q = (-1.5 \pm 2.2) \cdot 10^{-20} e$	1982	Bayreuth-ILL
magn. moment	$\mu = -1.91304308 (54) \mu_N$	1982	Harvard-ILL-Sussex- Rutherford
el. dipole moment	$d = (-1.8 \pm 2.9) \cdot 10^{-25} \text{ e} \cdot \text{cm}$	1985	
lifetime	$\tau = (937 \pm 18) \text{ sec}$ , direct meas. $\tau = (898 \pm 6) \text{ sec}$ , indirect meas.	1980 1985	Sussex-Rutherford
parity violating $\beta$ -asymmetry	$A = -0.1146 \pm 0.0019$	1985	Argonne-Heidelberg-ILL
ratio of weak coupling constants	$g_A/g_V = -1.262 \pm 0.005$	1985	
time reversal violating amplitude	$D = -0.0011 \pm 0.0017$	1976	ISN Grenoble-Yale
neutron oscillation period	$\tau_{nn} > 1 \cdot 10^6 \text{ sec}$	1985	CERN-ILL-Padova- Rutherford-Sussex
scattering amplitudes	np: $b = (3.741 \pm 0.004) \text{ fm}$ nd: $b = (-6.674 \pm 0.006) \text{ fm}$ nt: $b = (5.1 \pm 0.1) \text{ fm}$ etc.	1979	Vienna, Dortmund
parity viol. $\gamma$ asymmetry	np: $A_\gamma = (-4.7 \pm 4.7) \cdot 10^{-8}$ nd: $A_\gamma = (7.8 \pm 3.4) \cdot 10^{-6}$	1985 1984	ISN Grenoble Harvard
parity viol. spin rotation	$n^{117}\text{Sn}: \Phi/l = (36.7 \pm 2.7) \cdot 10^{-6} \text{ rad/cm}$	1982	Harvard-Rutherford-ILL- Ispra-Oak Ridge- Munich-Sussex
non linear term in free neutron Schrödinger eq.	$b < 3.3 \cdot 10^{-15} \text{ eV}$	1981	Bayreuth-Melbourne- Wien

# COLLEGE 4 STRUCTURAL AND MAGNETIC EXCITATIONS

## Members of the College

### I. Internal Members

L. Bernard  
R. Currat  
D. Degenhardt  
B. Dorner  
B. Farago  
P.H. Frings  
H.J. Lauter  
E. Lindley  
A. Magerl  
K. McEwen  
A.P. Murani  
R. Pynn  
B. Sarkissian  
H.P. Schildberg  
U. Steigenberger  
G. Steyrer  
W.G. Stirling  
Ch. Vettier  
K.R.A. Ziebeck  
C.M.E. Zeyen

### II. External Members

J.P. Boucher CEN-G  
G. Dolino USM-G  
C. Filippini CNRS  
F. Mezei Berlin  
L.P. Regnault CEN-G  
J. Rossat-Mignod CEN-G  
M. Vallade USMG

## GENERAL SUMMARY

As was already mentioned in last year's report, the experimental activity was severely curtailed because of the long reactor shut-down. This gave the opportunity for many of our colleagues to visit other research centres, where they carried out experiments and contributed to conferences, in particular to the International Conference on Neutron Scattering. Furthermore, certain members of College 4 have been involved in experiments or preparatory work related to the European Synchrotron Research Facility project, the proposed close collaboration with the ILL providing an unequalled research centre in condensed matter here. The scientific continuity of the college has, during this time, been maintained by the organization of weekly seminars. Finally, and when the ILL reactor resumed operation in September 1985, exactly on schedule, all College 4 instruments were operational and ready to recommence with the heavy scientific programme.

## SCIENTIFIC TRENDS AND HIGHLIGHTS IN 1985

Most of the experiments performed at ILL, under the auspices of College 4, and those conducted in 1985 by College 4 members at other centres, were devoted to the study of magnetic phenomena. This trend has been noticeable for

several years and was confirmed at the last meeting of the Scientific Council. It can be concluded, therefore, that neutron polarisation analysis will become an increasingly important feature of many triple axis spectrometers. Nevertheless, lattice properties are still of interest and especially in compounds of low-dimensionality or in materials exhibiting incommensurate phases.

### Lattice properties

One-dimensional inorganic conductors ( $\text{NbSe}_3$  and  $(\text{TaSe}_4)_2\text{I}$ ) which undergo Peierls instabilities and display non-linear electrical behaviour in the condensed state, have been investigated. The non-linear character of the dc-conductivity in these compounds is apparently associated with the electric-field unpinning of the current-carrying CDW (charge density mode, sliding mode). Although the structure of both compounds is well established, their lattice-dynamical properties have not been studied before due to the unavailability of single-crystal specimens of adequate size.

In the case of  $\text{NbSe}_3$ , a sample was prepared consisting of a large number of needle-shaped crystals assembled with a common  $\vec{b}$ -axis and random orientation around it. The spread in  $\vec{b}$ -axis orientations was found to be  $\sim 2.5^\circ$  f.w.h.m. Measurements near the strong (020) Bragg spot have yielded the value of the longitudinal sound velocity along the chain axis. A TA branch (of yet undetermined polarization) was also observed, via multiple-scattering processes. The  $(1, 2 \pm 2k_f, 0)$  satellite peaks were observed at 70 K, but inelastic constant-Q scans at these positions above  $T_p = 145$  K failed to reveal any phonon softening so far, due to a low signal-to-noise ratio (1).

In  $(\text{TaSe}_4)\text{I}$  superlattice reflections appear below  $T_p = 263$  K, at  $(h \pm 0.05, k \pm 0.05, l \pm 0.085)$ . The low acoustic dispersions at room temperature have been investigated (2) and a strong anisotropy was found between the two TA polarizations for wavevectors  $(\eta, \eta, 0.085)$ . At  $\eta = 0.05$  measurements as a function of temperature indicate some softening of the TA branch polarized in the basal plane.

### Magnetism

High energy magnetic excitations were observed in iron<sup>(3)</sup> with high energy transfers up to 320 meV, which is certainly a new record for IN1, the triple axis spectrometer on the hot source. The measured inelastic magnetic response agrees very well with recent theoretical predictions.

Normal rare earth (RE) metals are still being extensively studied. As well as excitations within the split ground state of RE ions (energies of a few tens of meV), it is possible, in principle, by neutron scattering techniques, to observe transitions between multiplets of different total angular momentum (energies of hundreds of meV). Intermultiplet transitions in the RE metals Praseodymium, Neodymium and Terbium have been studied during a visit to the IPN Source of the Argonne National Laboratory (4). Although samples were contaminated by hydrogen, some intermultiplet transitions have been identified but these experiments should be followed by measurements using purer samples and with improved resolution to study possible linewidth effects of the conduction electrons. Many other neutron scattering experiments on magnetic systems have been performed at neutron research centres, including Risø (Denmark) and Oak Ridge Laboratory (U.S.A.).

A new class of materials called heavy fermion systems, has been a focus of interest for the last few years. In such compounds f electrons are coupled to the conduction electrons and condense into a collective state of low temperature. They are characterized by an enhanced linear term in the low temperature specific heat, corresponding to large electronic effective masses. This implies that there is a narrow peak in the f electron density of state at the Fermi level. Many experiments are currently under way at ILL. They involve studies of crystal field excitations and quasielastic scattering in order to establish the nature and the energy scale of the spin fluctuations. Well known heavy fermions are being studied:  $CeCu_2Si_2$ ,  $CeCu_6$ ,  $UPt_3$ ,  $UBe_{13}$ . A new material  $CePb_3$  was recently (5) shown to order magnetically with a double incommensurate structure close to type I antiferromagnetism (Fig. 11). Such complicated structures have been observed in other Kondo systems. Inelastic scattering experiments have revealed broadened crystal field excitations at  $T = 100$  mK, whereas the quasielastic scattering is very weak. Such behaviour has to be understood in terms of different relaxation processes within the ground and excited states.

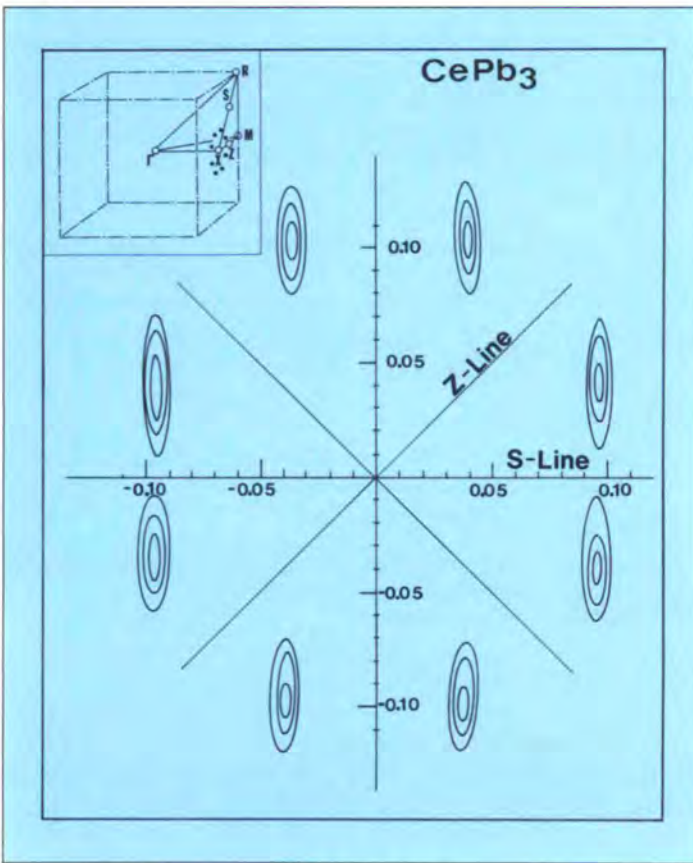


Figure 11: Intensity contour on the Brillouin zone surface around the x-point (0,0,1) in  $CePb_3$  at  $T = 500$  mK. These measurements indicate a doubly incommensurate structure, characterized by the wave vector  $(\mu_1, \mu_2, 1/2)$ , with  $\mu_1 = 0.135$ ;  $\mu_2 = 0.058$ . The apparent octagonal symmetry is purely accidental.

### Soliton excitations

During the past ten years the magnetic excitations of  $(CH_3)_4NMnCl_3$  (TMMC) have been studied by neutron scattering at several laboratories. For temperatures above  $T_N$ , at which three-dimensional spin ordering occurs ( $T_N \sim 2$ K for an applied field  $H \sim 4$ T), the manganese spins may be con-

sidered to belong to independent chains. The nearest neighbour exchange interaction along the chain is antiferromagnetic and local effects constrain the manganese spins to lie in the plane perpendicular to the chains. When a large magnetic field ( $6T \geq H \geq 2T$ ) is applied in the easy magnetic plane, soliton excitations are expected. In contrast to the ferromagnetic-chain case illustrated by  $CsNiF_3$ , the solitons in TMMC involve a rotation of the spins by  $\pi$  (rather than  $2\pi$ ) about the chain axis. The effect of these solitons - which can be thought of as moving domain walls - on the antiferromagnetic Bragg scattering was observed several years ago in a series of experiments on IN12. Both energy and wavelength broadening of the Bragg scattering was found to be in accord with theoretical expectations (6). In order to observe the solitons themselves, rather than their effect on the Bragg scattering, an experiment using neutron polarisation analysis has recently been carried out on IN12 (7). As shown in Fig.12 the external magnetic field  $\vec{H}$  ( $\sim 4.5$  T) was applied perpendicular to the scattering plane x-y, yielding a spin-flopped state with spins parallel to x. It is well known that neutrons are scattered only by the components of magnetic fluctuations which are perpendicular to the wavevector transfer  $\vec{Q}$  and that magnetic fluctuations parallel to  $\vec{H}$  induce non spin-flip scattering, whereas fluctuations perpendicular to  $\vec{H}$  induce spin-flip scattering. Thus the antiferromagnetic Bragg scattering is spin-flip while the solitons themselves scatter in the non-spin-flip channel. In order to observe the latter in the presence of intense Bragg scattering, full polarisation is required. The results obtained in the experiment are somewhat surprising as Fig. 12 demonstrates.

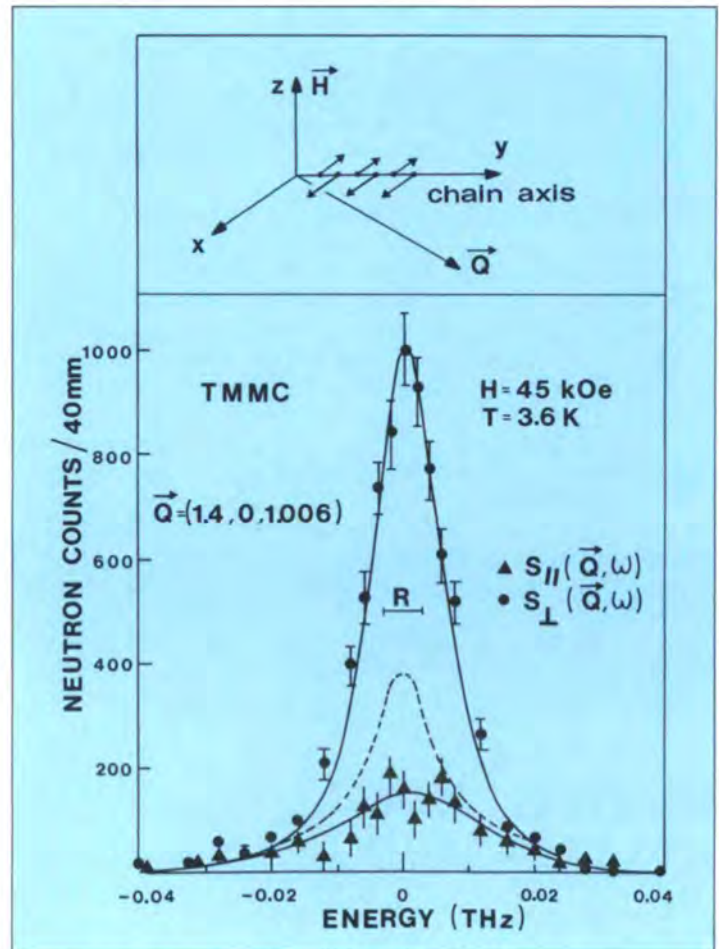


Figure 12: Bragg ( $S_{\perp}$ ) and soliton ( $S_{\parallel}$ ) scattering in TMMC. Inset shows the scattering geometry.

$S_{\perp}(\vec{Q}, \omega)$  in this figure is the spin-flip antiferromagnetic Bragg scattering which is evidently well described by the solid theoretical curve.  $S_{\parallel}(\vec{Q}, \omega)$  which is the non-spin-flip soliton scattering, isolated for the first time in this experiment, is, however, not described by the simplest theory (the dashed curve). In fact it is found that the effects of collisions between solitons and magnons and among solitons, causes both an energy broadening and a renormalization of  $S_{\parallel}(\vec{Q}, \omega)$ . A phenomenological calculation of these effects gives the lower solid line in the figure. However more theoretical work will be required if this subtle effect is to be understood properly.

## Synchrotron radiation experiments

In the context of the forthcoming ESRF installation in Grenoble, members of College 4 devoted their scientific activities to the synchrotron radiation. Several scientific meetings on the applications of X-ray synchrotron radiation were organized (8) and attracted distinguished groups of X-ray scattering and synchrotron radiation experts. Furthermore various aspects of X-ray scattering have been used as complementary tools to neutron scattering. These experiments involve inelastic X-ray scattering, surface scattering and magnetic X-ray scattering. They are described in the inserted box. It is hoped that the further use of both the neutron and synchrotron radiation techniques will become possible at Grenoble when the ESRF installations are operational.

Secretary: C. Vettier

## Experiments with Synchrotron Radiation carried out during the long Reactor Shut-Down

The complementarity of thermal neutrons from a reactor and X-rays from a synchrotron, or better, a storage ring, has been invoked several times. During the long shut-down of the ILL reactor some members of College 4 performed experiments with synchrotron radiation, thus proving the interest of neutron physicists in this relatively new technique.

In the following we recall some properties of neutrons and X-rays, not exhaustive, but rather introductory to the three experiments which will be presented. The wavelengths of both types of radiation are comparable to distances in condensed matter and allow an effective investigation of correlations in space leading to an understanding of the microscopic structure of condensed matter. The study of correlations in time on the other hand can be performed in two general ways: first, as it is traditionally done with neutrons, by measuring the Fourier transform by means of inelastic scattering, and secondly by observing the variation of the scattering properties of a sample in real time. As far as the first technique is concerned, the energy resolution in neutron scattering, nowadays  $\Delta E \leq 50$  neV, will never be reached in inelastic X-ray scattering (not considering coherent Mössbauer scattering). An experiment going to the limit of energy resolution for X-rays ( $\Delta E \approx 10$  meV) will be described below. The second technique of real time analysis is being used with neutrons as well as X-rays. In this case the resolution is much better for X-rays with  $\Delta t < 1$  nsec.

The interaction of the magnetic moment of the neutron with the magnetic moment of unpaired electrons is comparable with the nuclear interaction. Elastic and inelastic magnetic scattering of neutrons is, and will continue to be, probably the most important domain in research with thermal neutrons. For a long time it has been thought that neutrons are a unique probe of magnetic moments. Actually the cross-section for magnetic scattering of X-rays (a relativistic effect) is about 6 orders of magnitude smaller. But the advent of very intense and highly collimated X-ray beams from storage rings allows magnetic X-ray scattering experiments as described below.

The penetration into matter is another aspect, which reveals complementary applications of neutrons and X-rays. The high penetration of the "neutral" neutrons provide information of bulk properties, while the absorption of X-rays due to photoeffect and Compton scattering reduces the penetration considerably. This often unwanted restriction turns out to be an advantage for investigations of surfaces. Corresponding experiments will be presented below.

With the new intense sources of X-rays the challenge turned up to perform inelastic scattering similar to inelastic neutron scattering. Calculation i(1) showed that an energy resolution of about  $\Delta E \approx 10$  meV should be possible. Curved focussing crystals have to be used to obtain high enough intensity. Such a resolution will reasonably be applied to inelastic scattering with energy transfers  $\hbar\omega \leq 50$  meV.

This new technique overlaps with neutron scattering but provides several advantages. The resolution  $\Delta E$  stays the same up to energy transfers of several eV. Each scan at constant scattering angle is simultaneously a scan at constant momentum transfer  $Q$  and the momentum transfer can be kept

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- 6 L.P. Regnault, J.P. Boucher, J. Rossat-Mignod, J. Bouillot, W.G. Stirling, *J. Phys. C115*, 1261 (1981).
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small. The latter aspect has the advantage to reduce the unwanted background from multiphonon scattering which increases with a high power of  $Q$ . Furthermore small samples can be examined as the necessary volume lies below  $\text{mm}^3$  (for neutrons  $\text{cm}^3$ ).

Inelastic X-ray scattering is expected to be applied to different areas of research such as lattice dynamics of hard materials and materials containing light elements; molecular spectroscopy (with much smaller momentum transfer than with the Be-filter detector in neutron scattering), collective excitations in liquids (without the kinematic limitations due to the drastic change in wavevector connected with energy transfer in neutron scattering), dynamics of one surface (impossible with neutrons) etc.

A prototype spectrometer has been set up at HASYLAB, Hamburg, by the Sektion Physik, Prof. J. Peisl of München University with the cooperation of B. Dorner from the ILL. The high energy resolution is obtained by backscattering, similar to the instrument IN10 at ILL, but not quite the same way, because the X-ray beam, with the velocity of light, cannot be chopped effectively. For the X-ray case, the Bragg angles have to be kept slightly smaller than  $90^\circ$ . The energy transfer is scanned by varying the temperature of the monochromator crystal relative to the analyser, a technique applied on the backscattering instrument IN13 at the ILL.

So far the intensity was not high enough to observe real inelastic scattering. The resolution has been tested with a Pyrolytic Graphite crystal as well as with an amorphous scatterer,  $\text{SiO}_2$ -glass, at the sample position. The highest resolution obtained was  $\Delta E = 11 \text{ meV}$  with an  $E_0$  of  $13.8 \text{ keV}$  representing  $\Delta E/E = 8 \times 10^{-7}$  (2).

The cross-section for magnetic X-ray scattering is much smaller than that for charge X-ray scattering. The spin contribution to the scattered **amplitude** is reduced from the charge scattering **amplitude** by a factor  $\lambda_c/\lambda$  times the ratio of magnetic to total electrons, where  $\lambda_c$  is the Compton length,  $\lambda_c = \frac{h}{mc} = 0.0243 \text{ \AA}$ , and  $\lambda$  is the wavelength of the X-rays.

When there is no interference between the magnetic and the charge scattering amplitudes (in the case of non ferromagnetic materials), the **intensity** ratio is about  $5 \times 10^{-6}$ , a factor which can be made up for with a synchrotron radiation source. The first application of magnetic X-ray scattering, by De Bergevin and Brunel (3), was even made on a conventional X-ray tube, but with a prohibitively low counting rate. Further studies by Gibbs et al. (4) using synchrotron radiation have shown that such techniques are a very powerful tool. In the case of ferromagnetic compounds, there is an interference term in the scattered intensity, which contains the magnetic structure factor and the imaginary part of the usual structure factor. This term can be maximized by varying the incident X-ray wavelength near the absorption edges using the tunability of the X-ray energy from a synchrotron source. Such a method was used for the study of interfacial magnetism in superlattices  $\text{Gd}_n\text{Y}_m$  (5) at the Cornell University Synchrotron Source (CHESS) with the collaboration of Ch. Vettier as guest scientist from A.T.T. Bell Labs. and ILL.

These materials are composed of alternating regions of  $n$  magnetic Gd planes and  $m$  non-magnetic Y planes. There is considerable interest in the properties of interfaces between magnetic and non-magnetic materials and superlattices provide a means of studying an array of interfaces. A first study

has shown that the ferromagnetic component decreases on going from the centre of a Gd region towards the interfaces. No evidence was found for magnetic dead layers, with zero magnetic moment on Gd atoms, or antiferromagnetic coupling. These results were obtained on samples with rather large period ( $n = 21$ ,  $m = 21$ ), but measurements should be extended to samples with shorter period where the interfacial regions constitute a larger fraction of the sample.

Some experience was gained by H.J. Lauter during his stay at HASYLAB (Hamburg) concerning the application of X-ray scattering to physisorbed systems. Physisorbed layers can be investigated using, among other techniques, neutron and X-ray diffraction. Phases and phase transitions of many systems have been studied, mainly on exfoliated graphite. This graphite has a high surface area of 2 to  $20 \text{ m}^2/\text{gr}$  depending on the preparation. The main emphasis in X-ray diffraction studies has been on adsorbed heavy rare gases whereas studies using neutrons have concentrated on adsorbed light atoms and quantum systems. The crossover between the two techniques was in the region of the Ar-mass. The isotope  $^{36}\text{Ar}$  is particularly suited for neutron diffraction due to its exceptional high scattering cross section. Ar has, on the other hand, still sufficient electrons to give a good signal in X-ray diffraction. The presence of the form factor makes it nevertheless more difficult for the X-ray technique to study the higher order reflections of the adsorbed layers.

However the introduction of sophisticated storage rings like DORIS at HASYLAB (Hamburg) and the development of wigglers on these storage rings, led to more and more intense X-ray beams with narrow collimation and small wavelength spread. Thus, finally, a high resolution study of a physisorbed layer on a graphite single crystal (surface area about  $1 \text{ mm}^2$ ) has been possible and the technique of aligning the sample in the totally reflecting geometry results in the X-ray diffraction becoming only surface sensitive. Therefore, in particular, the reconstruction of single crystal surfaces can be studied. The new technique of using a single crystal surface as a substrate for a physisorbed system has previously not been possible by neutron diffraction and is unlikely to be in the near future.

Neutrons on the other hand, offer inelastic studies which give an insight into both the behaviour of the diffusion process and that of phonons in an adsorbed layer and thereby, the dynamical behaviour.

In conclusion, both techniques remain complementary methods of investigation but the very intense beams on storage rings have opened up the field of surface studies to an extent that cannot yet be envisaged with a neutron beam on a high flux reactor.

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# COLLEGE 5 CRYSTAL AND MAGNETIC STRUCTURES

## Members of the College

### I. ILL Members

J. Baruchel  
P. Bordet  
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F. Tasset  
P. Timmins  
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D. Waller  
S. Wilson  
A. Wright  
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C. Zeyen  
K. Ziebeck

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M. Backmann (CNRS)  
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D. Givord (CNRS)  
D. Gregson (CEN-G)  
J.-C. Marmeggi (CNRS)  
G. Roult (CEN-G)  
M. Schlenker (CNRS)  
Tran Qui Duc (CNRS)  
J. Vicat (CNRS)  
P. Wolfers (CNRS)

## GENERAL SUMMARY

Although there were few neutrons available in 1985 due to the long shutdown and major rebuilds or complete reconstruction of instruments in the diffraction group, some interesting work has been started and sometimes old data were found to be still exciting. A good part of the college activity however went into instrumental improvements or improvement in the environment such as the PS300 colour vector graphic display system, and it is hoped that from next year the user will find the full scientific advantage from the major effort put into technical upgrading. Instruments such as D2B, D9B, D19B and D20 are ready to set a new standard in their fields. To enhance the scientific interaction between the members of the college, weekly internal seminars were started this summer and are considered as a welcome addition to the existing exchange between college members and visitors. The college was considerably involved in the International Workshop on 2D-Position Sensitive Detectors and some members have used part of the reactor shut-down for a sabbatical leave, all helping to maintain the scientific interactions as lively as ever.

During 1984 several members of the college left Grenoble. P. Mangin finally returned to Nancy, first spending some months at NBS, Washington. R.F.D. Stansfield took up a post at the University of Edinburgh. A. Fitch will go to the University of Keele. We would like to thank all of them for their exceptional work as instrument responsables and scientists.

## WORKSHOP ON EVALUATION OF SINGLE-CRYSTAL DIFFRACTION DATA FROM 2-D POSITION SENSITIVE DETECTORS

Scientists from ILL, from the EMBL Grenoble Outstation, and from the LURE Synchrotron at Orsay organized an International Workshop on "Evaluation of Single-Crystal Diffraction Data from 2-D Position Sensitive Detectors", held at ILL 4-6 November, 1985.

The Workshop brought together some 80 specialists in the design, implementation and use of data evaluation techniques for a wide variety of 2-dimensional PSD's. These included multi-wire, film and TV detector systems for both conventional X-ray sources and high intensity synchrotron sources; detectors for long-wavelength and thermal neutrons, and for neutron time-of-flight instruments.

Fundamental aspects of the statistics of experiments were covered; and as well most of the algorithms used in working or planned software packages were discussed. These range from essentially statistical methods of peak detection, through maximum entropy methods, dynamic masking, profile learning, empirical fits, optimized-volume integration methods and fits based on resolution functions. The implications of new computer technology e.g. transputers, and of fast electronic hardware methods of data reduction were outlined.

The Proceedings of the Workshop will appear in early 1986 as a special volume of the Journal de Physique, Colloque Series. Further, it was agreed that a more specialized workshop would be organized in 1986, by Dr. G. Bricogne, in order to produce a sequence of modular, transportable routines of wide applicability to single-crystal 2-D PSD's.

## SCIENTIFIC HIGHLIGHTS IN 1985

### The Maximum Entropy Method (MEM)

Considered now as being one of the most general reconstruction techniques this method found its way into crystallography within the last few years. Members of the college were involved in this move and the applicability of MEM was studied both aiming at structure analysis and for the treatment of data from position sensitive detectors. The main part of the structure analysis consisted in the investigation of reconstruction of three dimensional images from three or more projections, and results on test data were encouraging. In the treatment of detector data a technique was developed for dealing with single Bragg reflections recorded on small position sensitive detectors. Smoothness and single-peakedness were included in the formalism, and simulation calculations showed the bias in intensity calculations of weak peaks to be negligible. An analysis of sensitivity showed that for peaks with an intensity of more than four times its standard deviations the method would detect correctly more than 50% of the peaks.

### Ab initio Structure Solution

In recent years members of the college have been engaged in the development of ab initio structure solution. Both high resolution neutron (DIA, ILL) and synchrotron (HasyLab) data

have been used. The main goal has been to estimate the limit of the method, and several known structures were used, notably  $\text{Ca}_5(\text{SiO}_4)_2(\text{OD})_2$ ,  $\alpha\text{SCOOH}$  and glycylglycine. The method is simply to extract from the spectra the individual Bragg intensities by a profile fitting method (G.S. Pawley, *J. Appl. Cryst.* 14 (1981) 357), followed by standard structure solution methods such as direct or Patterson methods. To ensure success the requirement is very high resolution, and the best obtained was for synchrotron data, where the full width of half height of the powder profile at focussing was  $0.03^\circ$ . Strain or particle broadening is clearly a problem, but in their absence it can be estimated that in general it should be possible in a routine manner to deal with structures containing around twenty atoms. At present more high resolution synchrotron data on unknown structures has been collected, and is being analysed. With the advent of the new D2B instrument more neutron data will be collected as well.

## Organic Superconductors

The first organic superconductors found belong to the family of isostructural salts  $(\text{TMTCF})_2\text{X}$  (with  $\text{C} = \text{S}$  or  $\text{Se}$ ;  $\text{X} =$  a small inorganic anion) [TMTCF is TMTTF if  $\text{C} = \text{S}$ ; Di(2,3,6,7-tetramethyl-1,4,5,8-tetrathiofulvalenium); TMTCF is TMTSF if  $\text{C} = \text{Se}$ ; Di(2,3,6,6-tetramethyl-1,4,5,8-tetraselenafulvalenium)]. In most of these compounds the HT metallic phase transforms to a low temperature (LT) ground state which is insulating non-magnetic (charge density wave (CDW) state), insulating antiferromagnetic (spin density wave (SDW) state), or superconducting (e.g.  $(\text{TMTSF})_2\text{ClO}_4$  below 1.2 K at ambient pressure). Furthermore the SDW ground state may turn to a superconducting state under moderately high pressure (HP) (e.g.  $(\text{TMTSF})_2\text{PF}_6$  for  $T < 1.2$  K and  $P > 6.5$  Kbars). The challenge is thus to make out the differences in behaviour of these salts which may aid in the synthesis of better organic superconductors.

The interplay of driving forces of the instabilities is currently understood mainly through theoretical models in which some major parameters are structural ones. Schematically, the electrical conductivity occurs mainly via the network of orbital overlap of chalcogenide atoms (C), thus a good knowledge of the relative influence of the nature of the "C" atoms, of the anion size and geometry, and of T and P induced changes in that network is essential. However, up to now structural data have been limited to room or moderately low temperatures at ambient pressure and calculations were based on questionable extrapolations of these data to lower temperatures and/or HP.

In a Bordeaux-ILL collaboration working on samples provided by the ORSAY group, a unique set of LT and/or HP structural data on some  $(\text{TMTCF})_2\text{X}$  salts (e.g. for  $(\text{TMTSF})_2\text{PF}_6$ ) has been obtained. The results have helped to clarify the rôle of the anion: e.g. the 24 K anion order-disorder transition in  $(\text{TMTSF})_2\text{ClO}_4$  does not lead to a metal-insulator phase transition since the corresponding distortion of the organic stacks is very weak; in  $(\text{TMTSF})_2\text{PF}_6$  no superstructure is associated with the anion freezing since the  $\text{PF}_6$  degrees of freedom are only progressively lost. They also showed that the above extrapolations are not satisfactory e.g. both T and P lead to a shortening in the ratio 1.5-2:1 of inter-versus intra-stack "C" atom interactions which enhances the 2D character of the interaction network. Furthermore modifications in the relative strengths and directions of the interstack interactions occur which make the LT electronic picture of these materials dif-

ferent from that believed up to now. These data have made possible a detailed quantum chemical approach to the transfer integrals and of the changes in the Fermi surface topography as a function of T and P (Fig. 13) in these materials. It is found that the wave vector is incommensurate in the SDW phase and a correlation is observed between the LT electronic state (i.e. superconducting or not) and the b-component of the Fermi surface nesting vector.

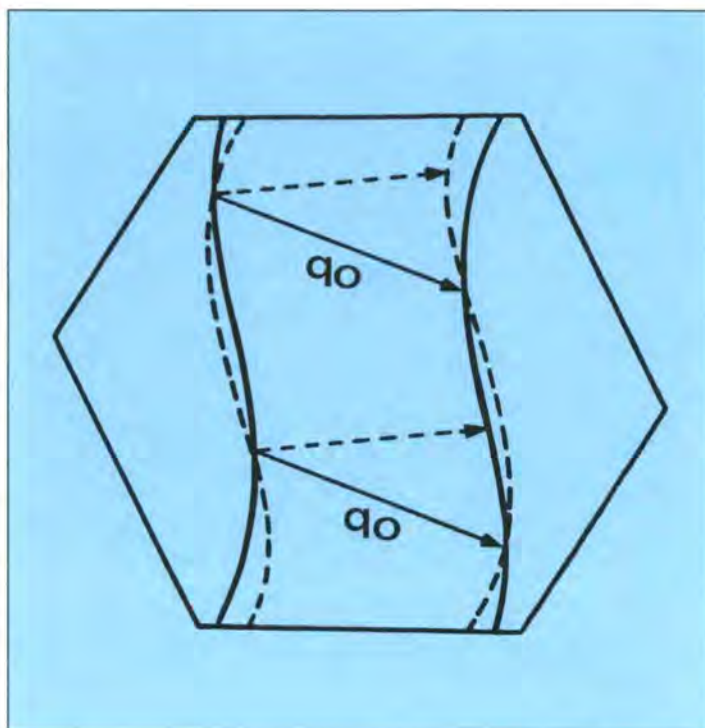


Figure 13: Fermi surface and optimal nesting vector  $q_0$  in  $(\text{TMTSF})_2\text{ClO}_4$ . Dashed lines:  $T = 300\text{K}$ ; full lines:  $T = 7\text{K}$ .

## Nuclear and Magnetic Extinction

Surprising results were obtained when measuring several sets of nuclear and magnetic satellite reflections in the helimagnetic phase of a crystal of MnP displaying a very severe extinction. For the  $2,0,0$  and  $2 \pm \zeta, 0, 0$  reflections, for instance, the magnetic reflecting powers  $\rho_{\pm}$  are twice as big as the nuclear one  $\rho_N$ , in spite of the fact that the scattering cross-sections  $|F_{\pm}|^2$  are more than ten times smaller than the nuclear  $|F_N|^2$ . The measurement of the reflecting powers as a function of crystal thickness (Fig. 14a to d) lead to an explanation for these very different nuclear and magnetic extinction factors for the same family of reflecting phases. The nuclear results in the helimagnetic phase appear consistent with dynamical theory (Fig. 14a). On the other hand, the magnetic scattering can be explained by using a model based on the known chirality domains (i.e. regions with right or left handed rotation of the helix) pattern. These domains divide the sample into optically incoherent slices while not affecting the nuclear diffraction (Figs. 14b and 14c), a situation which is similar to the so-called type II secondary extinction case. A similar explanation, based on domains, also holds for the ferromagnetic case (Fig. 14d). This domain related reduction of extinction for magnetic or mixed reflections was also observed, in a less spectacular way, in a wide variety of good quality magnetic crystals (MnP,  $\text{MnF}_2$ , YIG, Fe-3%Si, Tb, Ho ...).

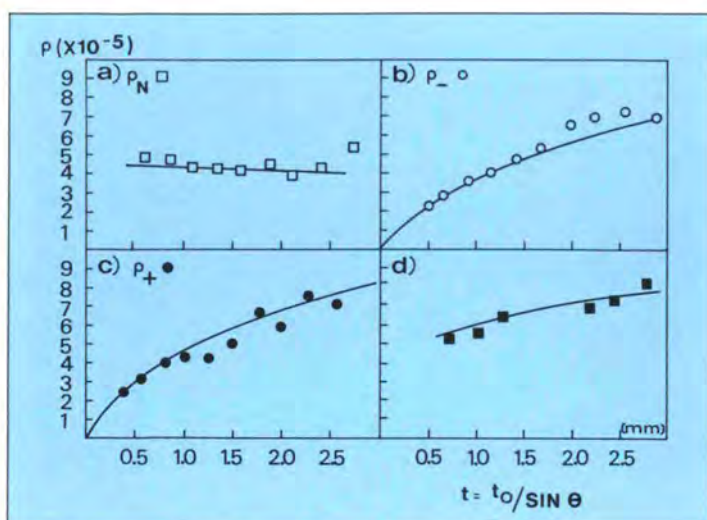


Figure 14: Reflecting power as a function of crystal thickness of MnP (see text).

## Neutron Topography

Polarized neutron topography allowed the observation of 180 type antiferromagnetic domains in  $\text{MnF}_2$  and  $\text{CoF}_2$ . A striking thermal memory effect has been observed on most of the samples: the domains basically remain unaltered after warming to  $T_N + \Delta T$  and then cooling below  $T_N$  if  $\Delta T < T_M$  (Fig. 15). The value of  $T_M$  was found to change from 0 to 50 K depending on the preparations of the  $\text{MnF}_2$  crystals. A strong correlation between this memory effect and the oxygen content of the crystals has been established by investigating the same samples using polarized neutron topography and luminescence techniques. This result supports the idea that most of the unexplained memory effects in antiferromagnetic domains, when no variation of distortion exists between neighbouring domains, could be impurity related ones. This was already suggested to explain experimental results in the case of chirality domains in the helimagnetic phase of terbium.

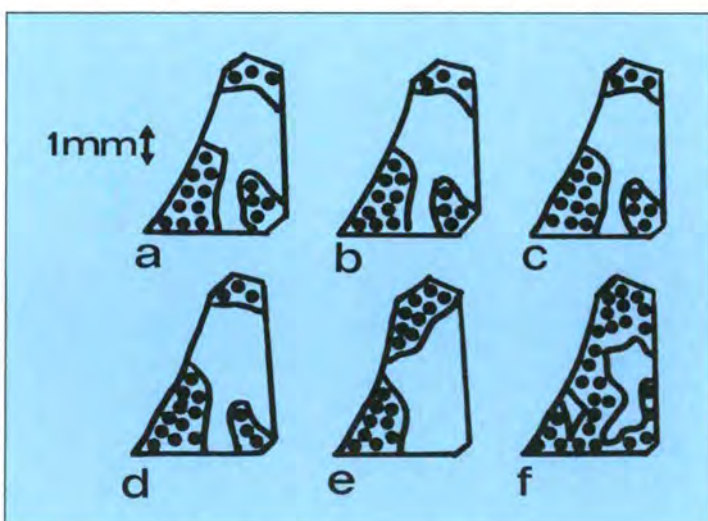


Figure 15: Domain pattern observed on topographs performed at 25K on a (001) platelet shaped crystal of  $\text{MnF}_2$ , using the 210 reflection and neutrons polarized along [001]. The topographs were recorded on X-ray film backed by a  $^{157}\text{Gd}$  converter (average exposure time = 8 hours). Sample cooled from a)  $T = 300\text{K}$ , warmed up to b)  $T_N + 30\text{K}$ ; c)  $T_N + 40\text{K}$ ; d)  $T_N + 50\text{K}$ , e)  $T_N + 60\text{K}$ ; f)  $T_N + 90\text{K}$  before being cooled again.

## Thermodiffractometry in Magnetic Systems

The good resolution of D1A and high flux of D1B have been used to investigate the two frustrated magnetic structures of the inverse Weberite  $\text{Fe}_2\text{F}_5(\text{H}_2\text{O})$ . This structure (Fig. 16) is built up from  $\text{Fe}^{3+}\text{F}_6$  and  $\text{Fe}^{2+}\text{F}_4(\text{H}_2\text{O})_2$  tetrahedron showing cornices and it presents a triangular arrangement of

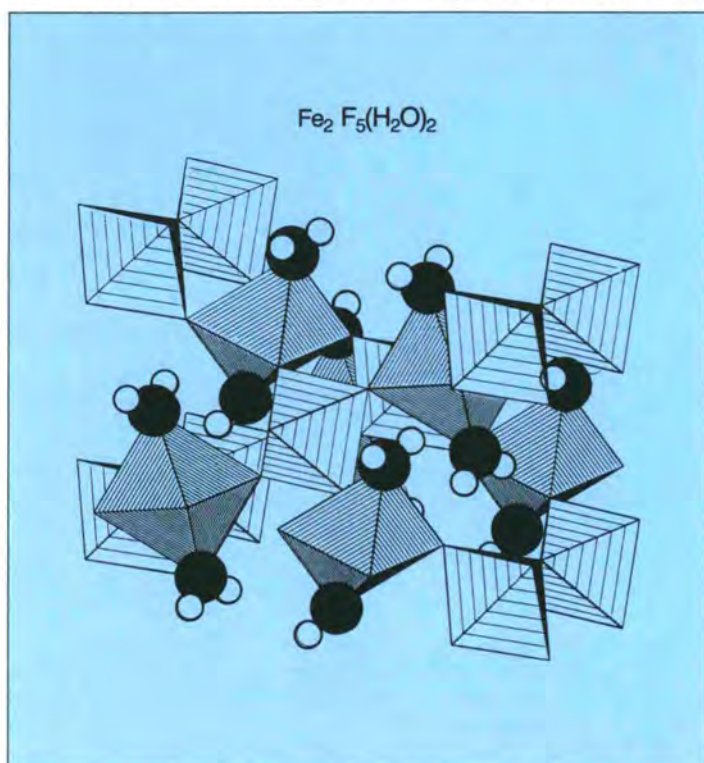


Figure 16: STRUPLO (R.X. Fischer, Mainz) representation of the inverse weberite  $\text{Fe}_2\text{F}_5(\text{H}_2\text{O})_2$  showing the linking of  $\text{Fe}^{3+}\text{F}_6$  (light) and  $\text{Fe}^{2+}\text{F}_4(\text{OH})_2$  (dark) octahedra. Oxygen and protons of the water molecule are represented by spheres.

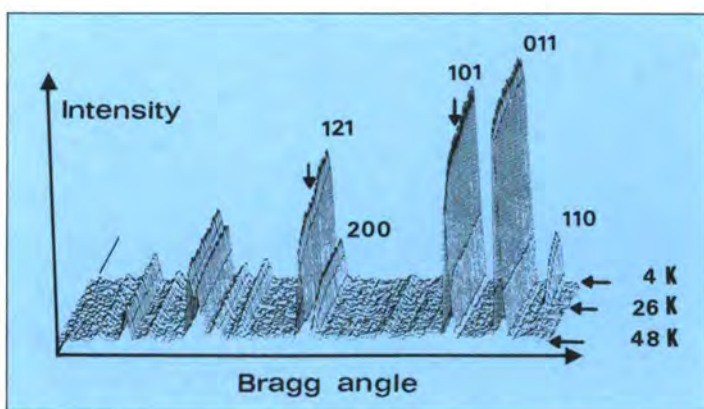


Figure 17 a): Thermodiffractogram of inverse Weberite  $\text{Fe}_2\text{F}_5(\text{H}_2\text{O})_2$  between 4K and 48K (note the change of intensity of some reflections at 26K).

magnetic cations  $\text{Fe}^{3+} - \text{Fe}^{2+} - \text{Fe}^{3+}$  propitious to frustration effects? A thermodiffractometric study on D1B (Fig. 17a) has shown the existence of two magnetic phase transitions at 48 K and 26 K respectively. The two magnetic structures were determined and refined (Rietveld profile refinement) from D1A data and lead to the following conclusions:

- between 48 and 26 K, the anisotropy of  $\text{Fe}^{2+}$  governs the magnetic structure and leads to an unexpected parallel arrangement of  $\text{Fe}^{3+}$  moments along the b axis while the canted

spins of  $\text{Fe}^{2+}$  lie in the  $b^*c^*$  plane.

- below 26 K the antiferromagnetic coupling between  $\text{Fe}^{3+}$  grows stronger and obliges the moment to adopt a different arrangement with a predominant antiferromagnetic component along  $c^*$ .

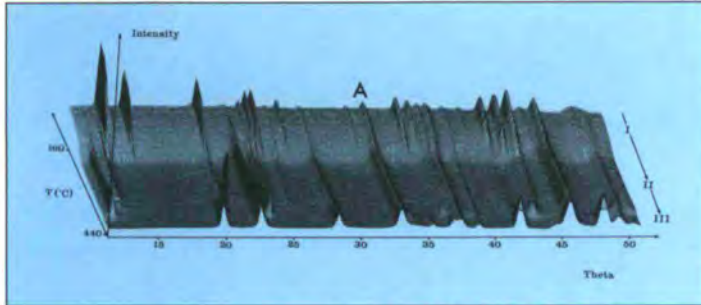


Figure 17 b): Thermodiffractogram of inverse Weberite in the temperature range 20-440 C°.

### Magnetic Phase Transitions and Spin Ordering in Iron Silicates

Magnetic behaviour in quasi-one dimensional systems is of considerable theoretical and experimental interest. A number of silicate minerals containing  $\text{Fe}^{++}$  or  $\text{Fe}^{+++}$  ions in edge sharing infinite single, double, etc. chains have been examined using high resolution neutron powder diffraction on D1A. For example  $\text{Ca}(\text{Fe}^{++}, \text{Fe}^{+++})\text{Fe}^{++}\text{Si}_2\text{O}_7\text{O}(\text{OH})$  ilvaite (Ghose, Hewat & Marezio (1984) Phys. Chem. Min. 11, 67) contains edge sharing double chains of  $\text{Fe}_A$  oxygen octahedra, infinite along the c-axis, with edge-sharing  $\text{Fe}_B$  octahedra alternatively above and below the plane of these double chains (Fig. 18). Below about 400 K, the electronic transition from an itinerant to an ordered state has been studied by calculating the effective valence charges, using precise Fe-O bond length measurements obtained from profile refinement of neutron powder diffraction data.

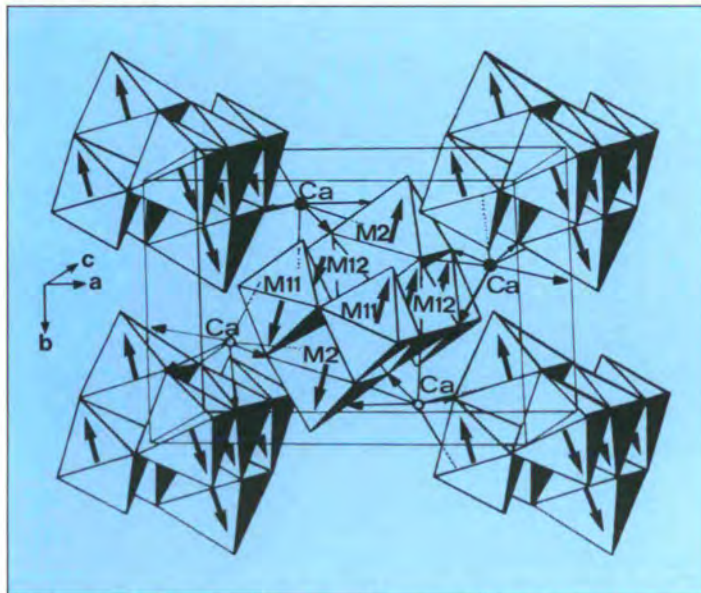


Figure 18: Magnetic structure from high resolution diffraction of ilvaite on D1A.

The  $\text{Fe}^{++}$  and  $\text{Fe}^{+++}$  ordering on the A-sites can be clearly demonstrated. It has been shown that below 116 K the  $\text{Fe}^{++}$  and  $\text{Fe}^{+++}$  spins on the A-sites along one infinite c-axis chain are parallel, and antiparallel to those along the adjoined edge sharing centro-symmetrically related site. Below 40 K,

this order is maintained, but the  $\text{Fe}^{++}$  spins on the B-sites order antiferromagnetically as well.

A similar pattern of ferromagnetic ordering within chains, plus antiferromagnetic ordering between chains has been obtained for orthoferrosilite  $\text{Fe}_2\text{Si}_2\text{O}_6$  (Fig. 19, Hewat, Ghose & Van Dang).

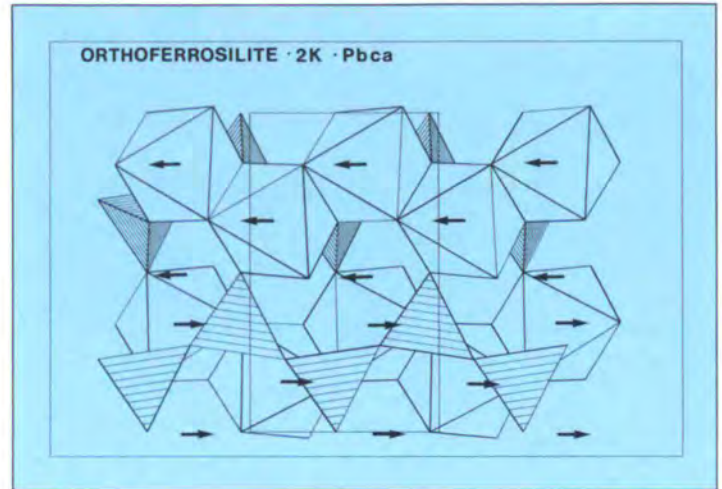


Figure 19: Ferromagnetic ordering within chains plus antiferromagnetic ordering between chains in orthoferrosilite.

Precise D1A powder measurements of the low temperature monoclinic structure of magnetite  $\text{Fe}_3\text{O}_4$  (Hewat, Marezio & Ghose) have clearly shown the monoclinic splitting at the Verwey transition (Fig. 20a) and the weak superlattice lines due to the ordering of  $\text{Fe}^{++}$  and  $\text{Fe}^{+++}$  (Fig. 20b). Valence

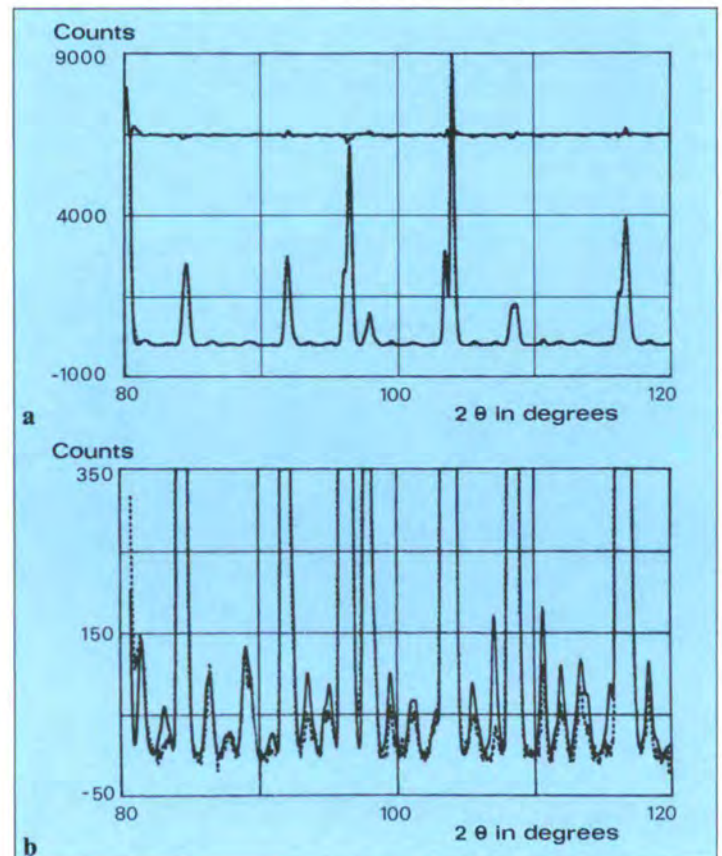


Figure 20: Powder measurements of the low temperature monoclinic structure of magnetite. (a) monoclinic splitting at the Verwey transition, (b) weak superlattice lines due to the ordering of  $\text{Fe}^{++}$  and  $\text{Fe}^{+++}$

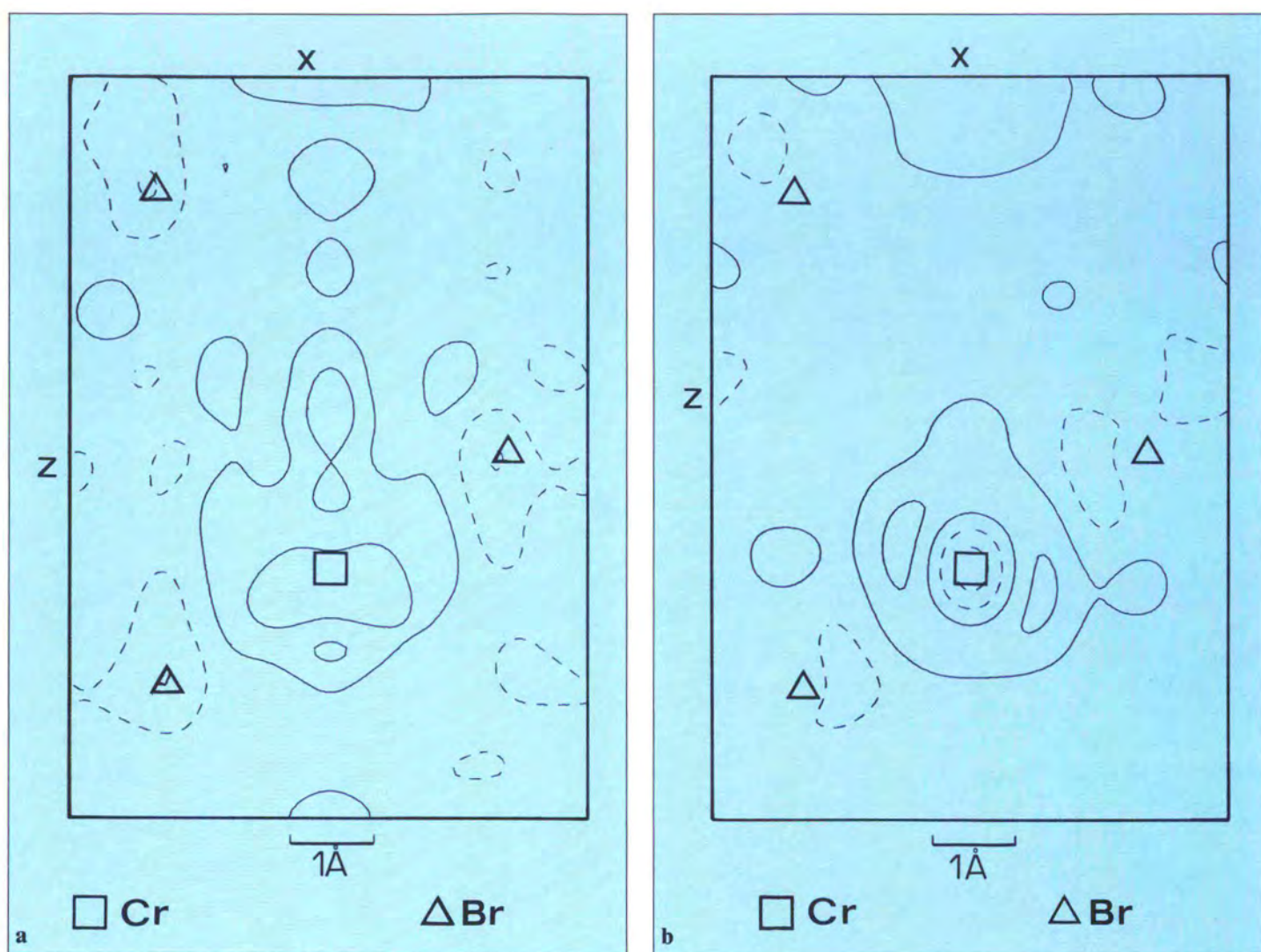


Figure 21: Spin density measurements in  $\text{CrBr}_3$ . (a) covalent spin density obtained from polarized neutron measurements. (b) corresponding fitted density.

charge calculations from precise Fe-O bond lengths obtained by profile refinement have identified some of the  $\text{Fe}^{++}$  sites, but others, related by a pseudo centre of symmetry, have not yet been unambiguously determined. The new very high resolution powder machine D2B should permit a complete solution of this problem.

### Covalent Spin Density in $\text{CrBr}_3$

The period of the long shut-down has been used to further develop a 'convolution' technique for modelling the spin density in crystals. The technique is particularly appropriate in systems in which the wave-functions of the magnetic atoms or ions mix with those of their ligands. In such systems the spin density contains contributions from both 'atomic' and 'overlap' terms. The special feature of the convolution technique is that it allows calculation of both the atomic and the overlap contributions to the magnetic structure factors from a model wave function in such a way as to allow a straightforward least-squares fit of the parameters of the model to the measured structure factors.

The method has been applied to the ionic ferromagnet  $\text{CrBr}_3$ . Figure 21a shows the 'covalent' spin density (that not modelled by a single chromium ion calculation) obtained from

polarised neutron measurements; Fig. 21b shows the corresponding fitted density. The parameters obtained from the fit indicate that most of the observable covalency effects are in the orbitals with  $A_1$  symmetry. The minority spin density at the bromine sites being due to spin polarization of the doubly occupied bonding orbitals, the diffuse ( $4s$ ) density around the chromium site is due to the bonding orbital.

Secretaries: W. Kuhs  
A. Hewat

## THERMAL DIFFRACTOMETRY

Even in the earliest days of neutron scattering the temperature was one of the parameters most frequently varied. Indeed, because of the good transmission of neutron beams through most materials, variable temperature diffraction experiments are often much more easily carried out with neutrons than with X-rays. It is however only recently, aided by the advent of reliable position sensitive detectors and computer piloted temperature controllers, that it has become practical to plot the detailed evolution of diffraction patterns as a function of temperature. This new technique which has been called thermal diffractometry promises to rank alongside differential thermal analysis and calorimetry providing a powerful new tool for solid state chemistry.

Thermal diffractometry is carried out on a powder diffractometer equipped with a wide angle position sensitive detector, such as the instrument D1B at the ILL. With such an instrument the whole of a powder diffraction pattern, out to the angular limit of the detector, is collected simultaneously, there being no need to scan the detector in steps through a range of scattering angles. Instead the temperature, or some other intensive parameter, is varied in steps and a complete diffraction pattern collected at each step. The results of such a scan can be displayed as a "thermo-diffractogram" such as that illustrated in Fig. 17b. This figure shows the thermal decomposition of the inverse Weberite  $\text{Fe}_2\text{F}_5 \cdot 2\text{H}_2\text{O}$ . The experimental results are displayed as a three-dimensional picture of the diffraction pattern. The horizontal axis in the plane of the paper gives the scattering angle and the vertical axis the diffracted intensity. The variation with temperature is displayed in perspective along the third axis. A Bragg reflection which varies little as a function of temperature appears as a ridge such as that labelled A in Fig. 17b. At a phase transition such a ridge may split up or disappear and new ridges corresponding to Bragg reflections of the new phase will appear. In Fig. 17b three stages in the decomposition can be distinguished. In stage I dehydration leads to the formation of three crystalline phases identified from the diffraction pattern as  $\text{FeF}_2$ , HTB- $\text{FeF}_3$  and Pyr $\text{FeF}_3$ . During this stage a continuous reduction in the background is observed as the diffuse scattering from hydrogen diminishes as water is driven off. In stage II the Pyr $\text{FeF}_3$  transforms to HTB- $\text{FeF}_3$  with the resulting increase in height of the ridges corresponding to this phase. Finally in stage III the HTB- $\text{FeF}_3$  transforms to the stable form of iron trifluoride R- $\text{FeF}_3$ .

The study of the crystallisation of amorphous alloys provides a further example of the application of thermal diffractometry. Indeed a "thermo diffractogram" showing the crystallisation of the amorphous alloy  $\text{Fe}_{80}\text{P}_{10}\text{C}_{10}$  appears as Fig. 22 of the 1984 Annual Report. The diffractogram shows clearly the three stages in the crystallisation process. At low temperatures the only feature is the broad hump at low angle due to short range order in the amorphous state. As crystallisation begins the peaks characteristic of  $\alpha$ -iron appear, finally in the crystalline state both the  $\alpha$ -iron peaks and those from the intermetallic compounds  $\text{Fe}_3\text{P}$  and  $\text{Fe}_3\text{C}$  are seen.

In cases such as that of crystallisation, where an irreversible reaction is studied, valid results can only be obtained using a technique by which the whole diffraction pattern can be recorded simultaneously, and only when the neutron source is sufficiently intense to allow a statistically significant count to be obtained in a time less than the characteristic time of the reaction.

# High Resolution Neutron and Synchrotron Powder Diffraction

A. W. Hewat\*

\* Based on an invited paper at the French & Swedish Academies of Science meeting in honour of Professors Guinier and Hägg, Stockholm, 8-9 May 1985. To be published in *Chemica Scripta*

## INTRODUCTION

In the 1960's, but for Professor Bertaut, powder diffraction might have been ignored when planning the high flux Grenoble reactor. In 1985, very high resolution and high flux powder diffractometers occupy one of the prime neutron beams at the ILL, while a powder diffractometer is the biggest machine on the new SNS pulsed neutron source near Oxford. American laboratories, such as Argonne, Los Alamos, Brookhaven, NBS-Washington and Missouri, are all building new powder diffractometers to compete with the Europeans. The future is also bright for high resolution X-ray powder diffraction, with the promise of tunable wavelengths from the European Synchrotron source to solve unknown structures by anomalous dispersion, and new detectors for chemical kinetics. The European Synchrotron will strongly complement the neutron diffractometers, but conventional X-ray laboratories will also be stimulated by these new machines, just as the new interest in neutron powder diffraction has contributed to the renewal of medium flux reactor centres.

With over 1000 papers published in this area within the past ten years, it is instructive to see that the fields of magnetism and phase transitions, where neutrons and powder diffraction are uniquely suited, are still most strongly represented, but new areas such as zeolites and solid electrolytes are growing fast. In fact, a rapidly increasing percentage of studies are on real materials which have eventual industrial applications, or even immediate relevance as in the case of the experiments on stress in welds or prestressed wires, objects which are too large for other non-destructive measurements.

## THE GROWTH OF HIGH RESOLUTION POWDER DIFFRACTION

The development by Rietveld (1967) of a method of refining crystal and magnetic structures directly from their neutron powder diffraction pattern (1) (2), without the usual step of extracting 'structure factors', is the most important reason for the rapid development of neutron powder diffraction in

recent years. Until 1973 these methods remained little known outside two or three reactor laboratories (Rietveld, Loopstra, Van Laar, Andresen et al, references cited by Cheetham and Taylor, (3)). From 1973 they were taken up by many outside users of neutron diffraction.

Figure 22 shows the resulting rapid increase in the number of papers published in this area. Most of the neutron work was based on the original Rietveld computer program with some modification (4)-(7) (Hewat 1973; Sabine and Clarke 1977; Wiles and Young 1981; Bendall, Fitch and Fender 1983). Independent profile refinement programs were also developed, often from established single crystal programs (8)-(11) (Taylor and Cox 1973; Klein and Weizel 1975; Will 1979; Pawley 1980; Matthewman et al 1982).

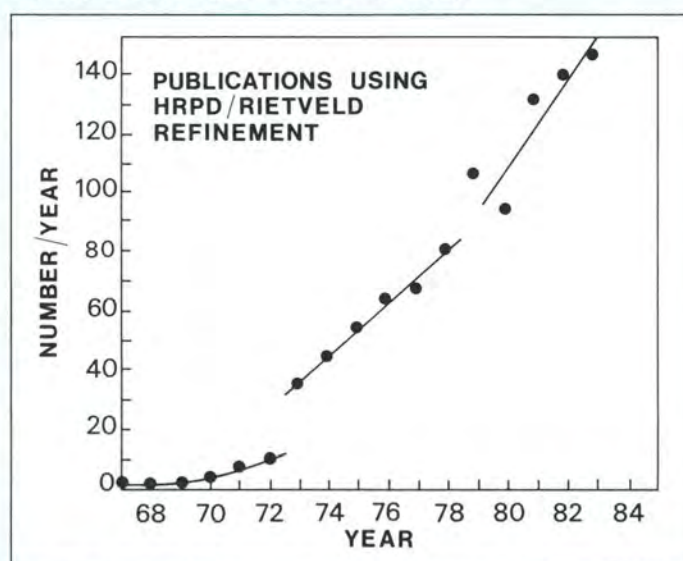


Figure 22: Number of publications using high resolution powder diffraction or Rietveld refinement showing rapid increases with the dissemination of the techniques and the construction of new diffractometers.

The number of publications was further stimulated from 1975 by the development of new high resolution powder diffractometers freely available to external users (12)-(16) (Hewat 1975; Carpenter, Mueller et al 1975; Hewat and Bailey 1976; Jorgenson and Rotella 1982; Howard et al 1983). New profile refinement programs were developed for the time-of-flight (TOF) machines (17) (von Dreele, Jorgensen and Windsor 1982).

From 1977, ten years after the original neutron work of Rietveld (1967), the method was adopted for X-ray diffraction (18)-(23) (Malmros and Thomas 1977; Young, Mackie and von Dreele 1977; Werner, Marinder and Magneli 1978; Werner, Salome and Malmros 1979; Kirik et al 1981; Baerlocher 1984). Each of these developments has produced new growth in the number of publications.

## CLASSIFICATION OF RESEARCH USING HIGH RESOLUTION POWDER DIFFRACTION

The more than 1000 papers can be grouped under the 20 subjects shown in Table 1. The total in each category is also given, with the number in the single year 1984 given in paren-

theses. This latter figure illustrates that some fields, such as zeolites and electrolytes, are growing more rapidly than older areas such as magnetism and structural transitions.

The first column in Table 1 lists those areas for which powders have natural advantages over single crystals. For example, materials which undergo phase transitions often break up on cooling, or at least form multi-domain crystals less suitable for conventional crystallographic study - the sample is more naturally available in powder form. In particular, solid liquids and gases are easier to prepare and handle as powders. Other materials, such as zeolites, may be only available as powders and some materials may break up on intercalation. Catalysts are usually powders, and the purification process of crystal growth may be undesirable for the study of solid electrolytes (ionic conductors and electrode materials). Other defected or disordered structures and non-stoichiometric compounds may also be changed by single crystal growth. The superconductor category refers to materials such as partially oxidised Chevrel phases, where the transition temperature is sensitive to the stoichiometry. Finally, 'ceramics' includes those other oxides, nitrides etc. which are usually produced in powdered ceramic form, for example as dielectrics.

The second column in Table 1 lists powder diffraction subjects which are particularly suited to neutron diffraction. Magnetic structures are of course the prime example, since unlike X-rays, neutrons see the magnetic moments on the atoms. Because the neutron scattering factor is of similar magnitude for all nuclei, heavy metal compounds such as transuranics on the one hand, and hydrides and hydrogen bonds on the other, usually require neutron diffraction. Metal hydrides of course have potential applications for hydrogen gas storage. The study of other types of bonding, and of mixed valence compounds, may also be better done with neutron powder diffraction. This is simply because systematic errors in X-ray powder diffraction are more important, reducing the precision with which bond lengths can be determined.

The third column of Table 1 lists other subjects for which powders have been used with advantage, ranging from mineral structures to polymers and biological materials. High pressure structures can sometimes best be done with powders, and of course systematic measurements, as a function of

temperature, may be less time-consuming with powders.

Finally, we have a category for texture, stress and strain measurements. This is a well established subject for X-rays, but is rapidly acquiring new applications with neutron powder diffraction; the greater penetrating power of the neutron permits non-destructive testing of the interior of objects of cm<sup>2</sup> dimensions - welds, pressure vessels, rolled and worked metal, prestressed composite superconducting wires etc.

For the present purposes it is sufficient to refer to a few of the most recent examples in each of these categories. These papers themselves contain early references, but a complete bibliography will appear elsewhere (24) (Hewat and Cheetham 1986). Earlier reviews (3), (25)-(27) have been published by Cheetham and Taylor (1977) and Hewat (1980), with specialised reviews on ionic conductors by Santoro (1983) and on techniques and performance by Taylor (1985).

## RECENT RESULTS (A) HIGH RESOLUTION POWDER DIFFRACTION

Structural transitions in dielectric perovskites were among the early uses of profile refinement (reviewed by Hewat (4)), so it is not surprising to see papers such as that on PZT by the Japanese Ito, Shiozaki and Sawaguchi (1983) continuing to appear (28): the structures are important for understanding the dielectric properties, and difficult to obtain by other means. Powder diffraction has effectively taken over the study of structural transitions in many similar inorganics, with work on oxides and halides, such as NaBiTiO<sub>3</sub> by Vakhrushev et al (1983) in Russia (29), CaSnF<sub>6</sub> by Mayer, Reinen and Heger (1983) in Germany (30), NH<sub>4</sub>NO<sub>3</sub> by Ahtee et al (1983) in Finland (31), NH<sub>4</sub>AlF<sub>4</sub> by Bulou et al (1982) in France and (NH<sub>4</sub>)<sub>2</sub>SnBr<sub>6</sub> by Armstrong et al (1983) in Canada (33). Structural transitions involving ammonia are, of course, particularly well suited to neutron diffraction, as are those involving the cyanide group (KCN-KBr), studied extensively by Rowe et al (1984) in the USA (34).

TABLE 1

### Classification of Research Using High Resolution Powder Diffraction 10 Year Publication Total 1974/85 (1984 Total)

POWDERS	NEUTRONS	OTHER
87 ( 7) Structural T/Ns	150 (20) Magnetic structures	23 ( 7) Minerals
14 ( 5) Solid liquids, gases	59 ( 5) Heavy metal compounds	8 ( 1) Polymers & biological materials
40 (16) Zeolites	52 ( 8) Hydrides & H <sub>2</sub> storage	47 ( 6) Other structures
6 ( 0) Intercalates	32 ( 1) Hydrogen bonding	19 ( 4) High pressure
11 ( 4) Catalysts	3 ( 1) Other bonding	18 ( 2) Thermal expansion
81 (15) Solid electrolytes	7 ( 1) Mixed valence	20 ( 1) Texture, stress
73 (10) Order-disorder/defects		
7 ( 1) Superconductors		~ 150 Techniques
33 ( 4) Ceramics		21 ( 2) Synchrotron papers
36 ( 2) Other X-ray work		

10 Year Total (1984 total).

The problem of obtaining single crystals is often more difficult in the case of solid liquids or gases. Much earlier work (35) (36) was done in this area by Sandor et al and Koski (1975). More recently, Meier and Helmholdt (1984) at Petten studied solid oxygen, Powell et al (1984) at Chalk River solid halogens, Moodenbaugh et al (1984) at Brookhaven hydrogen sulphate and Overell, Pawley and Powell (1982) in Edinburgh a number of simple hydro- and halo-carbons such as carbonyl sulphide (37)-(40). One of the more active subjects recently has been the study of various forms of ice, usually at high pressure, by Jorgensen et al (1984) at Argonne (41) and Kuhs and Finney (1984) at Grenoble (42).

Another very active field in 1984 for neutron diffraction has been the study of zeolites, long a favourite subject for X-ray powder diffraction, which continues today (43)-(45) with, for example, the work of Baerlocher (1984) McCusker (1984) and Cartledge, Meir et al (1984). With neutrons, Adams et al (1984) have done much work in Grenoble on different zeolite-A structures with adducts such as carbon monoxide (46); Cheetham et al (1984) have similarly worked on zeolites -A, -Y and ZK4, investigating Si/Al ordering, which is difficult to do with X-rays, and studying chemical activity such as hydrolysis (47). One of the most beautiful examples of the power of neutron powder diffraction for looking at hydrocarbons in zeolites has been the study of benzene in Y-zeolite by Fitch et al (1985) in Grenoble (48) (see figure 23). Parise et al (1984) have recently published several papers in this area, for example on the presence of non-framework alumina in de-aluminated zeolites (49). There is at present a legal dispute about patent rights priority between US chemical companies (50); neutron diffraction can help distinguish Si from Al in the determination of the composition of proprietary zeolites.

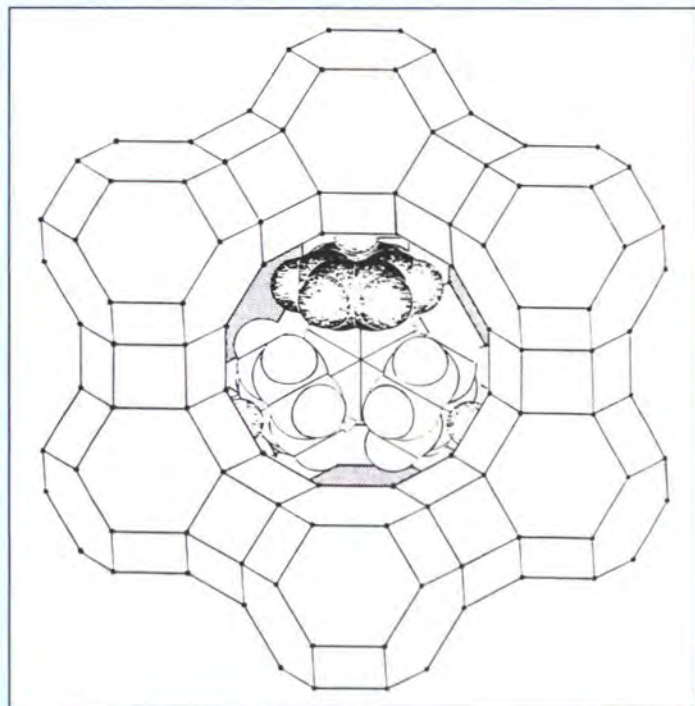


Figure 23: The four benzene positions and the sodium ions within the supercage of sodium Y-zeolite  $\text{Na}_{56}\text{Si}_{136}\text{O}_{384}\text{Al}_{56}$  (Fitch, Jobic and Renouprez<sup>48</sup>) from neutron powder measurements on DIA.

Zeolites are a particular class of materials which have otherwise been classed as intercalates and catalysts. Tachez et al (1982) have studied the intercalation of water into vanadyl

phosphate (51), and much low resolution work has been done on intercalation into graphite (52). Intercalates of hydrogen and  $\text{Li}^+$  will be treated separately under hydrides and solid electrolytes.

Various oxidation catalysts such as iron-bismuth-antimony molybdenum oxides are particularly suited to neutron powder diffraction (53)-(57). (Theobald et al 1984, 1985; Teller et al 1984, 1985; Laarif et al 1984). These materials are often characterised by close-packed oxygen lattices with periodic 'stacking faults' the details of which are difficult to investigate with X-rays in the presence of the heavy metal atoms.

Lithium insertion compounds are a particularly important class of 'intercalates', but have been classed with electrolytes, which includes ionic conductors and battery electrode materials. Because of the difficulty of locating light atoms, they have been natural candidates for neutron diffraction since the earlier X-ray work of (58) Werner, Marinder and Magneli (1978) on  $\text{Li}_{1-x}\text{Ta}_x\text{O}_{8x}\text{F}_x$  and Nord and Thomas (1978) on  $\text{LiTa}_3\text{O}_8$  (59). Hodeau, Marezio et al (1984) have studied the high temperature structure of  $\text{H-LiTa}_3\text{O}_8$  (60) following earlier work on  $\text{LiSn}_3\text{O}_8$  and  $\text{LiZr}_3\text{O}_8$ , while Cava, Santoro et al (1984 a,b) have published many papers (61) (62) on  $\text{Li}^+$  inserted heavy metal oxides, in particular those related to the Chevrel phases  $\text{Li}_3\text{Mo}_6\text{O}_8$ . Other recent  $\text{Li}^+$  insertion studies (63)-(66) include the metal oxides  $\text{Li}_x\text{RuO}_2$  and  $\text{Li}_x\text{IrO}_2$  by Davidon and Greedan (1984),  $\text{Li}_x\text{MoO}_2$  by Cox et al (1982),  $\text{Li}_x\text{NiO}_2$  by Orman and Wiseman (1984) and  $\text{Li}_x\text{CoO}_2$  by Thomas, David and Wiseman (1985). Brun, Jorgensen et al (1982) have used TOF neutron diffraction to look at the fast-ion electrode lithium-aluminium (67).

Even for the traditional lead-acid battery, surprisingly little is known about the solid state chemistry of the  $\text{PbO-PbSO}_4$  system. Lead oxide is of course a remarkably versatile material, which apart from being able to exist in different forms itself, can accommodate other ions in the space normally occupied by the lone pair electrons. Again the system has been largely neglected by X-ray diffraction because of the heavy atoms. Gavarrri et al (1981, 1985) have continually contributed to powder diffraction work in this area (68) (69), while Mentzen et al (1984) have started to explore the crystal chemistry of the  $\text{PbO-PbSO}_4$  system (70). Jorgensen et al (1982) have used neutron diffraction to explore the hydrogen content and lead deficiency in beta- $\text{PbO}_2$  itself (71). D'Antonio and Santoro (1980), and more recently Santoro et al (1983), have been working in this area (72) (73); Hill (1985) has published several papers on the subject, relating the structural information to the hydrogen loss concept of lead-acid battery failure, and reviewing the subject (74) (75).

Neutron work on hydrogen ion conduction is well illustrated by a series of papers (76) by Fender's group on hydrogen uranium phosphates and arsenates (HUP) where the  $\text{H}^+$  is shunted between hydronium ions in hydrogen bonded sheets. Rebbah, Pannetier and Raveau (1982) and Rotella et al (1982) have used neutrons to localise hydrogen in  $\text{HTiNbO}_5$  and  $\text{HTaWO}_6$  respectively (77) (78), while Wiseman and Dickens (1973) and Dickens and Weller (1983) have studied hydrogen-conducting bronzes  $\text{H}_x\text{WO}_3$  and  $\text{H}_x\text{ReO}_3$ ; this is similar to the work on  $\text{Li}^+$  conductors (79) (80).

The traditionally hydrogen, lithium, sodium, ammonium and silver beta-alumina ionic conductors (81)-(84) have been explored by Tofield et al (1979), by England, Jacobson et al (1982), by Tofield et al (1979) and by Newsam, Cheetham et al (1983). The more complex Na beta'-alumina (86) has been examined by Jorgensen, Rotella and Roth (1981). More

esoteric  $\text{Na}^+$  conductors, based on the  $\text{NaCl-UCl}_6$  system (87)-(89) have been discussed by Bogacz et al (1980), Hewat et al (1984) and Bendall, Fitch and Fender (1983). Much earlier low resolution work was done on silver conductors, with the most recent paper by Newsam et al (1984) on  $\text{Ag}^+$  conduction in  $\text{Ag}_3\text{PO}_4$  (90).

Finally, heavy metal oxide conductors (91)-(93) include  $\text{Bi}_2\text{O}_3$  by Verkerk et al (1982) and Battle (1983) following the work of Harwig (1978). Halide conductors (94) (95) have been studied by Lucas et al (1981) and Lucat et al (1980).

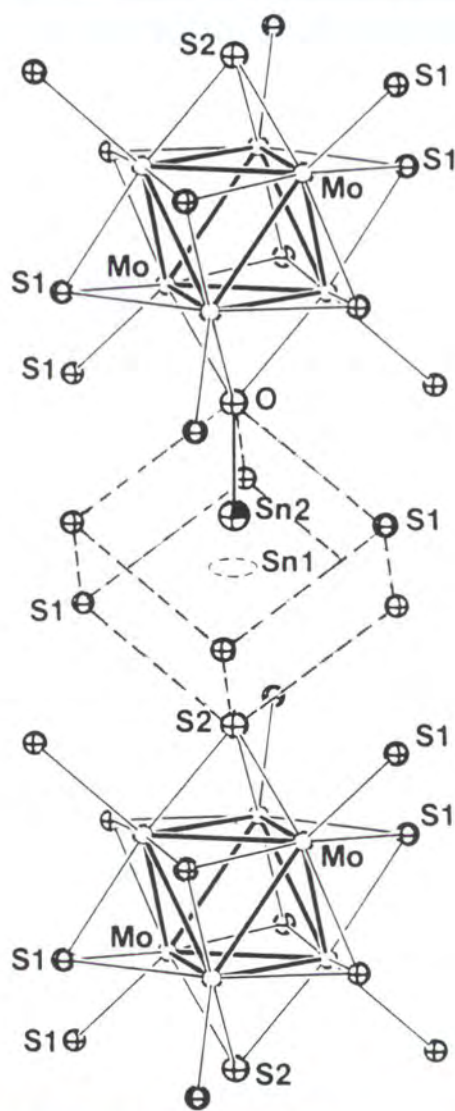


Figure 24: The structure of the oxygen defect in the Chevrel-phase superconductor  $\text{SnMo}_6\text{S}_8$  (Hinks, Jorgensen and Li<sup>107</sup>) from accelerator based TOF neutron powder measurements.

Other disordered and non-stoichiometric structures have long been the subject of neutron powder diffraction before the use of Rietveld methods (96). The metal oxide block structures (97) examined by Anderson et al (1975) are a particularly interesting example, similar in some ways to the heavy metal oxide catalysts. The application of X-ray and neutron methods to these problems was reviewed (98) by Cheetham (1981). Very high resolution diffractometers are particularly useful for such large block structures as  $\text{W}_3\text{Nb}_{14}\text{O}_{44}$  (99).

Obradors et al (1983) have looked at cation disorder in the ferrite  $\text{BaFe}_4\text{Ti}_2\text{O}_{11}$  (100). Nord et al (1984) have published many papers on cation disorder in mineral structures (101)-(103), while other recent work has included that of Wechsler et al (1984) on titanomagnetites and Wilkinson et al (1984) on wustite. Many of the recent papers on defected and disordered structures can better be classed under ionic conduction.

Non-stoichiometry has recently been of interest in the study of the depression of the superconducting transition in the Chevrel phases (106)-(108)  $\text{PbMo}_6\text{S}_8$ ,  $\text{YbMo}_6\text{S}_8$  and  $\text{SnMo}_6\text{S}_8$  (Fedorov et al 1982; Hinks et al 1984). Jorgensen and Hinks (1985) have described a model for oxygen impurities and low temperature structural distortions in these important materials (figure 24). Sato et al (1983) have also found structural transitions in superconducting  $\text{M}_x\text{WO}_3$  compounds (109).

Finally, we may group a number of publications on other oxides and nitrides under 'ceramics', this being the form in which they are produced for possible applications in magneto-electro- or optical devices (110)-(113). For example, Cadee (1983) has described the structure and luminescence of ceramic oxides such as  $\text{Pb}_3\text{GeGa}_{10}\text{O}_{20}$ . Fischer and Waldner (1982) have compared neutron diffraction and epr results on piezo-electric  $\text{Bi}_4\text{Ge}_3\text{O}_{12}$  and  $\text{Bi}_4\text{Si}_3\text{O}_{12}$  while Devreugd et al (1984) with  $\text{Ba}_4\text{ZrRu}_3\text{O}_{12}$  and Zandberg et al (1984) with  $\text{Ba}_3\text{SrRu}_2\text{O}_9$  have recently published several neutron powder refinements of the structure of ceramics. Jacobsen et al (1981) with  $\text{Ba}_3\text{Te}_2\text{O}_9$  has long been interested in oxides related to the perovskite structure (14).

## RECENT RESULTS (B) NEUTRON POWDER DIFFRACTION

The use of neutron powder diffraction in magnetism has experienced periods of growth but also of decline. There has been renewed interest recently with the development of very high resolution diffractometers using long wavelength neutrons to resolve modulated magnetic structures (115) (116) (figure 25).

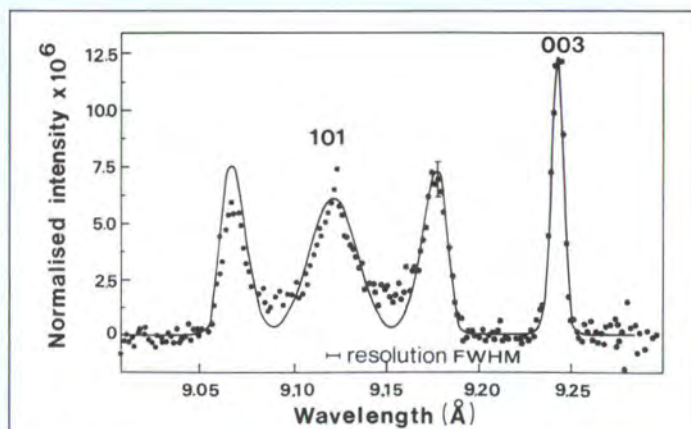


Figure 25: The 101 reflection and satellites from the  $620\text{\AA}$  spiral magnetic structure of bismuth ferrite (Sosnowska, Peterlin-Neumaier and Steichele<sup>116</sup>) from reactor based TOF measurements.

Work on magnetism is somewhat hampered by a lack of texts describing systematic methods of determining magnetic structures from indexed powder diffraction patterns. Bertaut (1963) has reviewed the application of group theory to the classification and determination of magnetic structures in general (117), and Prandl (1976) has applied irreducible representation theory to the garnet structure (118). A more empirical approach has been used by Ghose et al (1984) to determine the magnetic structure of the ferro-silicate ilvaite (119), a complicated monoclinic structure containing one  $\text{Fe}^{+++}$  and two  $\text{Fe}^{++}$  ions in each of four equivalent positions.

Recent work on magnetic structures (120)-(125) includes several papers on rare earth beryls ( $\text{ReB}_{13}$ ) by Vigneron et al (1985) and many on rare earth silicides and germanides ( $\text{ReCo}_2\text{Si}_2$  by Yakinthos et al 1984 and Leciejewicz et al 1984;  $\text{ReM}_2\text{Si}_2$  and  $\text{ReRh}_2\text{Si}_2$  by Slaski et al 1983;  $\text{ReCu}_2\text{Ge}_2$  by Schobinger - Papamentellos et al 1984;  $\text{Re}_2\text{Fe}_3\text{Si}_5$  by Moodenbaugh et al 1984). Helmholdt et al (1984) have reported on several Heusler compounds (126), while Herbst et al (1985) have recently produced several papers on  $\text{Re}_2\text{Fe}_{14}\text{B}$  and  $\text{Er}_6\text{Fe}_{23}$  (127) following work (128) by James et al (1983). The magnetic structures and properties of these  $\text{Re}_6\text{M}_{23}$  compounds have recently been reviewed (129) by Crowder and James (1983).

Battle et al (1984) have done much recent work on rare earth oxides in the series  $\text{Ba}^{++}\text{Re}^{+++}\text{Re}^{++++}\text{O}_6$  and on chromium phosphates and molybdates (130) (131). Nord (1983) has published  $\text{NiCoP}_4\text{O}_{12}$  and  $\text{NiZnP}_4\text{O}_{12}$  (132). Gavarri et al (1983) have papers on  $\text{MnSb}_2\text{O}_4$  and  $\text{NiSb}_2\text{O}_4$  (133) (134). Finally, Renaudin et al (1983), Courbion et al (1984) and Ferey et al (1985) have been working on ferrous fluorides  $\text{Ba}_2\text{FeF}_6$ ,  $\text{LiMnFeF}_6$  and  $\text{NH}_4\text{Fe}^{++}\text{Fe}^{++}\text{F}_6$  (135)-(137).

Heavy atom structures are again ideally suited for neutron diffraction, since unlike X-rays, neutrons have similar scattering lengths for all atoms. Loopstra (1970) used Rietveld's technique for his study of beta- $\text{U}_3\text{O}_8$  (138), followed by Taylor and Wilson (1974) and Levy et al (1975) who pioneered the use of Rietveld refinement for the structures of many such transuranic oxides and halides (139) (140). Von Dreele et al (1975) with their work on  $\text{Pr}_7\text{O}_{12}$  introduced Rietveld techniques to the USA (141). Recent work in this area (142)-(146) includes Ijdo (1982) on scandium sulphides, Harsta, Rundqvist and Thomas (1983) with several papers on the tungsten carbide system, Parise et al (1983) on  $\text{La}_3\text{WO}_6\text{Cl}_3$  tungstate, Christensen and Hazell (1984) on  $\text{Ho}_2(\text{OH})_4\text{CO}_3$  and Jones et al (1984) on yttrium-holmium dicarbides. The astonishing 'tour de force' of Zachariasen in this area, having only minute quantities for X-ray diffraction in the 1940's, has now been made much easier by the use of Rietveld refinement of neutron powder data.

At the other end of the atomic table, but equally suitable for neutron diffraction for the same reasons, are the hydrides and hydrogen storage materials. This is an area in which Scandinavian and Swiss laboratories have been particularly active, with several papers (147)-(150) including a review by Andresen (1982), publications on hydrogenated palladium phosphide by Andersson et al (1984), on  $\text{YD}_2$  and other metal hydrides by Fischer et al (1983) and work on  $\text{NiD}_2$  by Noreus and Werner (1984). The latter authors have also contributed to a number of recent publications (151)-(153) on  $\text{Mg}_2\text{NiH}_x$  and  $\text{Mg}_2\text{FeH}_6$  (Noreus and Werner 1982;

Didisheim et al 1984; Hayakawa et al 1984). Yartys et al (1983) and Bradhurst (1983) have reviewed the structural chemistry of these intermetallic hydrides and their use for hydrogen fuel storage (154) (155).

Neutrons have long been used for the study of hydrogen bonding, again particularly in Scandinavia. The earliest use of the Rietveld method in this area was for a study of the phase transition in ammonium di-hydrogen phosphate (156). Christensen et al (1977) have published a number of papers on  $\text{CrOOH}$  and  $\text{FeOOH}$ , and Adams et al (1981) studied several perhydrated structures, using difference Fourier techniques to locate the  $\text{H}_2\text{O}_2$  groups (158). Most recent work (159) (160) includes molybdic acid by Crouchbaker and Dickens (1984) and oxalic acid at high pressure by Putkonen et al (1985).

Neutron powder diffraction has advantages over its X-ray counterpart for the study of other types of bonding too. This is because the bond lengths are usually determined with greater precision, so that they have real chemical significance; Bertaut (1975) gives an early review (161). A typical recent example of the use of neutron powder diffraction is provided by Hathaway et al (1984) in a study of Jahn-Teller effects in monoclinic  $\text{Cu}(\text{OH})_2(\text{NH}_4)_2(\text{SO}_4)_2$  Tutton's salt (162). The bond lengths can be precisely measured as a function of temperature, showing a smooth transition from the expected elongated  $\text{CuO}_6$  octahedron at 5K to the flattened octahedron at 300K. The neutron experiment also revealed the onset of hindered rotation of the  $\text{NH}_4^+$  groups, which may be as important in driving the transition as any local Jahn-Teller effect on Cu(II). This paper provides an interesting internal check on the validity of the calculated standard deviations, since there are three independent water molecules, and H-O bond lengths can be compared at seven different temperatures. The calculated standard deviations, at least for this rather good data, appear to be as reliable as those for ordinary single crystal refinements.

Accurate measurement of bond lengths is also important for determining the effective valence in metal oxides and halides according to the rules (163) of Brown and Shannon (1973). It is particularly interesting in the case where the same atom species co-exists, in two valence states. The classical example is  $\text{Fe}_3\text{O}_4$  magnetite ( $\text{Fe}^{+++}\text{O}_3\text{Fe}^{++}\text{O}$ ), which undergoes a metal insulator transition when the  $\text{Fe}^{++}$  and  $\text{Fe}^{+++}$  ions order below 110K. Marezio has published several papers in this area, the most recent example of which (Ghose et al 1984) describes the ordering of  $\text{Fe}^{++}$  and  $\text{Fe}^{+++}$  in the iron silicide, Ilvaite (119): the precision obtained for the bond lengths is such that the  $\text{Fe}^{++}$  and  $\text{Fe}^{+++}$  sites are clearly distinguished. Prassides et al (1985) examined a number of mixed valence metal salts  $\text{A}_2\text{MX}_6$  (165), while Ferey et al (1985) have looked at pyrochlore  $\text{NH}_4\text{Fe}^{++}\text{Fe}^{+++}\text{F}_6$  using similar techniques (166). Other examples include  $\text{LaCoO}_3$  by Thornton et al (1982, 1985) which had previously been supposed to undergo such a transition, from  $\text{R}\bar{3}\text{C}$  to the lower  $\text{R}\bar{3}$  on heating (167) (168)! Jorgensen et al (1983) have used TOF neutron diffraction to look at the valence of ytterbium in their study of the Chevrel phase  $\text{YMo}_6\text{S}_8$  (169).

The study of minerals has always been a favourite subject for powder diffraction, and it is not surprising to see that neutrons are also contributing here, especially for measuring the magnetic structures of minerals. Fuess (1982) has reviewed this area (170), and recent examples (171) (172) include studies of garnets by Pieper, Fuess et al (1983) and her-

cynite by Hill (1984). The SYNROC (synthetic rock) materials have recently been proposed as alternatives to glasses for immobilising radioactive wastes. Sabine and Hewat (1982) have examined the stability of the hollandite component at high temperature (173), and Post et al (1982) have looked for cation mobility in hollandite, cryptomelane and priderite (174). The most recent work on hollandite (175) is that of Cheary et al (1984). Clay minerals (kaolinite, dickite) have received attention (176) (177) by Adams (1983) and Suitch and Young (1983). These materials are typical of layered compounds which pose a particular problem for Rietveld refinement in that lines are broader for scattering perpendicular to the layers than within the layers. Recent attempts have been made to model this effect (178).

Sample problems have also restricted the application of powder techniques to polymers (179) (180), which in any case require constraints to limit the number of parameters (Immirzi 1980; Young et al 1980). Applications to biological materials (181) (182), such as apatite tooth enamel (Jacquet et al 1980; Suitch et al 1985) often suffer from similar problems; in apatite the aim is to understand ionic transport and ordering, such as the effect of fluoride on tooth enamel.

High pressure measurements were once supposed to be particularly well suited to neutron powder diffraction because the great penetrating power of the neutron permits more massive pressure cells. Unfortunately this has not been realised in practice because the relatively low neutron flux also means more massive samples. Diamond anvil cells can be used to produce much higher pressure in very small X-ray samples. Even so, the neutron time-of-flight machines have some advantages because background scattering can be almost entirely eliminated with  $90^\circ$  scattering, as we saw for the ice VIII experiment at Argonne (41) compared to a similar study at Grenoble (42). Other experiments, such as those of Nelmès et al (1978) on  $\text{KH}_2\text{PO}_4$  needed neutrons to study hydrogen bonding effects under pressure (183). New types of pressure cell ((164) Vettier et al 1985) should increase the use of neutron powder diffraction for high pressure structure refinement, as opposed to the usual phase diagram studies with X-rays.

Finally, one high resolution neutron powder diffraction field which has been growing very rapidly does not involve Rietveld refinement. This is the non-destructive testing for stress and texture in bulk materials, for which X-rays have access only to the surface. By using  $90^\circ$  scattering and collimating the neutron beams,  $\text{mm}^3$  volumes can be examined within  $\text{cm}^3$  samples. Recent examples (184)-(186) include texture analysis using a PSD by Bunge et al (1982), the work of the Harwell group on welds (Slattery and Windsor 1983) and on macroscopic stress in steel plates (Allen et al 1985). Microscopic stress due to grain interaction in zircalloy (187) has been examined at Argonne (MacEwen et al 1983), while other macroscopic stress measurements (188)-(190) include Schmank and Krawitz (1982), Pintschovius et al (1983) and Kjems (1984). A particularly interesting example is described in a series of papers (191) by Flükiger et al (1983). They have investigated the superconducting properties of  $\text{Nb}_3\text{Sn}(\text{Cu})$  wires subjected to stress in large electromagnets (figure 26). The effects of a low temperature stress-induced phase transition can be reduced by prestressing the wire bundles with steel. Many of these bulk studies are not possible with other methods, and have immediate commercial applications.

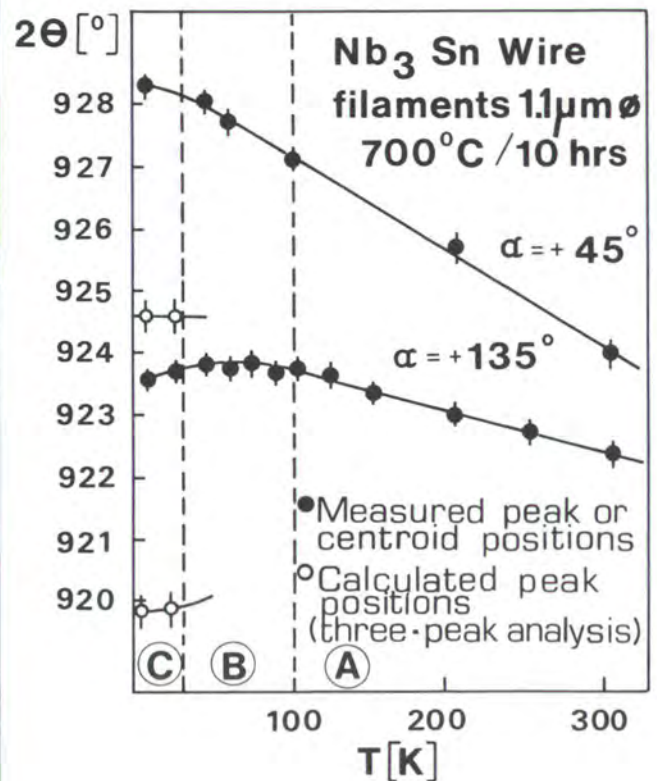
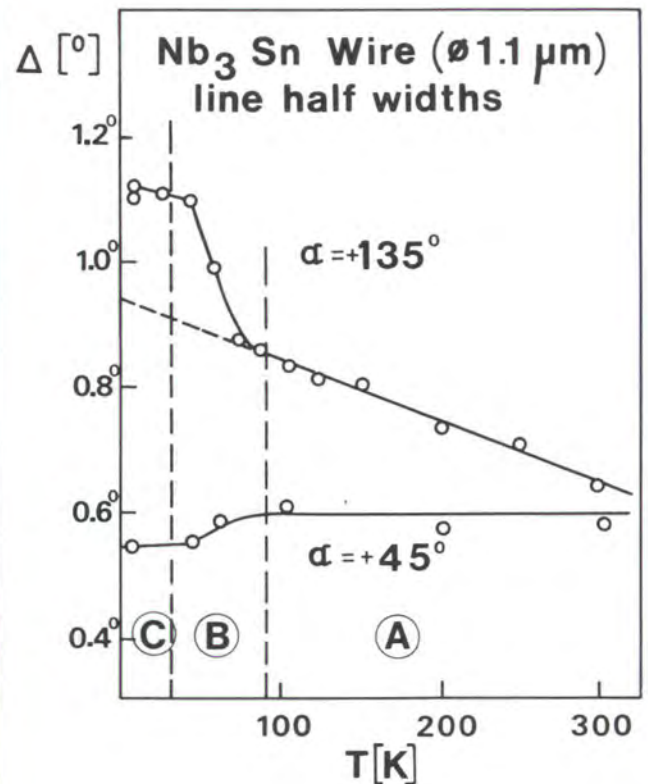


Figure 26: The temperature dependence of the (400) line width and position for different orientations of a  $\text{Nb}_3\text{Sn}$  multifilamentary superconducting wire (Flükiger et al<sup>191</sup>) from DIA measurements.

## THE FUTURE OF HIGH RESOLUTION NEUTRON POWDER DIFFRACTION

The rapid growth in high resolution powder diffraction and Rietveld refinement over the past ten years, shown in Figure 40, served mainly to develop the user base for the techniques and to explore new applications. The next few years will see the development of several new diffractometers and the use of profile refinement as a standard and common technique.

At present, the highest resolution machines, D1A on the ILL reactor and GPPD on the Argonne pulsed source, are comparable in both intensity and resolution. Table 2, from (192) Jorgensen et al (1985), uses two numbers to compare these very different machines - the resolution  $\Delta d/d$  and the product of the time averaged flux on the sample  $\Phi_s$ , the sample volume  $V$ , and the detector solid angle  $\Omega$ . The backscattering time-of-flight geometry of the GPPD permits a larger detector solid angle without loss of resolution, but the focussing monochromator of D1A delivers a larger time averaged flux to the sample.

D1A and GPPD are also used for similar types of experiments, and in practice produce similar results (193) (figure 27). The Argonne machine does have an advantage for high pressure work, and the Grenoble machine for indexing large structures.

The next generation of machines, D2B on the ILL Grenoble reactor, and HPRD on the ISIS pulsed source near Oxford, offer significant advantages in both resolution and intensity. These new machines will eventually have comparable performance (Table 2) but will do complementary science.

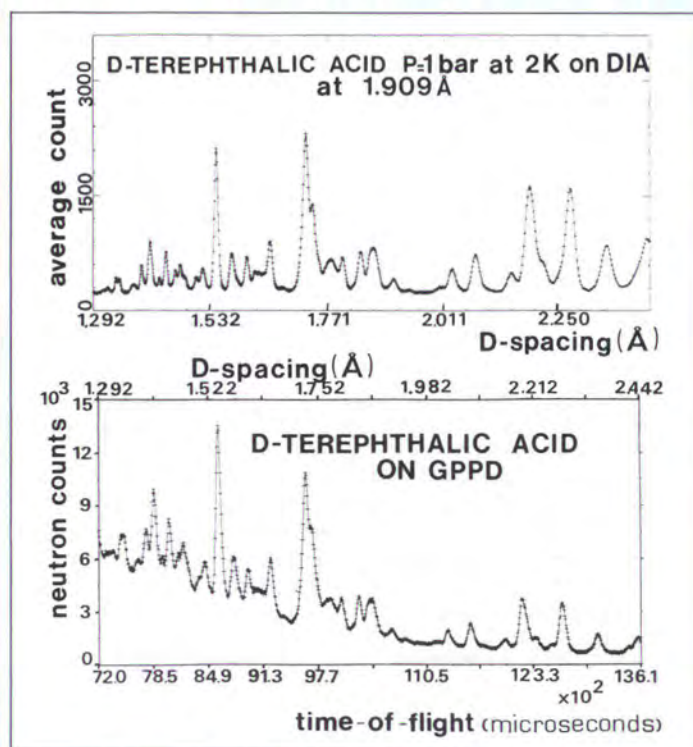


Figure 27: Similarity of reactor based (D1A) and accelerator based (GPPD) neutron powder diagrams of hydrogen ordering in terephthalic acid (Fischer et al<sup>191</sup>). Only a small section of the total pattern is shown.

In the USA a new TOF machine is also nearing completion on the Los Alamos pulsed source; it will have only medium resolution but very high intensity, and is probably best compared to the new high flux machine D20 also nearing completion at ILL (Pannetier, this meeting). There are also plans for building new high resolution machines at Los Alamos, Brookhaven and Oak Ridge to try to match the new European machines.

There are a number of medium to high resolution machines under construction or improvement on medium flux reactors (at Missouri, NBS-Washington, Lucas Heights, Paris, Studsvik, Würenlingen etc.). At ILL, as elsewhere, the main constraint is manpower and finance, not neutron beams - there are several spare neutron guide positions such as those occupied by D1A/D1B. There is a proposal (Steichele 1984) for a high resolution TOF machine on the second cold source (194).

## A HIGH RESOLUTION SYNCHROTRON POWDER DIFFRACTOMETER

Two types of powder diffractometer have been used with synchrotron X-ray sources: a high flux medium resolution machine (195)-(197) using an energy dispersive position sensitive detector for chemical kinetics (Buras et al 1979; Bourdillon et al 1978; Glazer et al 1978), and a high resolution classical diffractometer (198)-(200) pioneered at Brookhaven for profile refinement of crystal structures (Cox 1985; Hastings et al 1984; Cox et al 1983). Separate machines are required for these two purposes, corresponding to the separate neutron machines D20 and D2B at ILL. Such machines are already being developed at Daresbury and HasyLab (figure 28).

It has been said that synchrotrons produce so much intensity that single crystals the size of powder grains can be used. This may be true 'en principe' but in practice the only known example of this, a 6 micron  $\text{CaF}_2$  study (201) by Bachman et al (1985) is not entirely convincing.

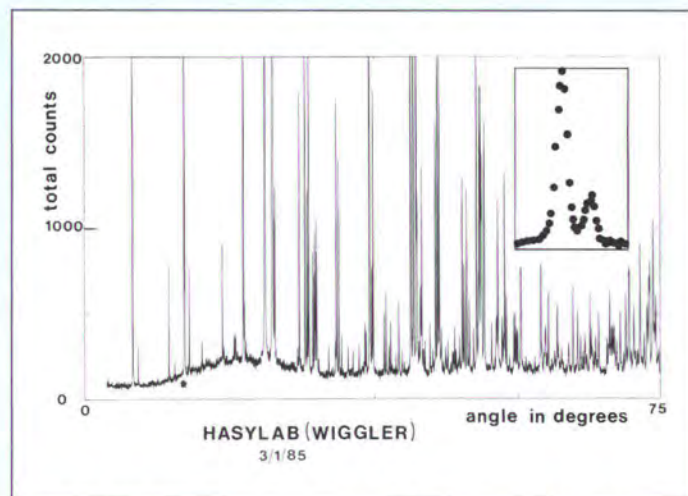


Figure 28: Synchrotron powder pattern for  $\text{Al}_2\text{Y}_4\text{O}_9$  using a small linear detector at HasyLab. The inset is the peak marked with an asterisk, and the steplength is 0.006°. (Christensen, A.N.; Feidenhansl, R.; Fjellvåg, H.; Lehmann, M.S.; Nielsen, M. and Pedersen, J.S. - personal communication).

It may also be argued that neutron powder diffractometers are now so good, reaching the resolution limits fixed by powder particle size, that X-ray synchrotron machines are simply not needed. This is to ignore the advantages of the different types of radiation: neutrons see all of the atoms equally well, which is an advantage for structure refinement but a disadvantage for structure solution. In future, structure solution and not just structure refinement will be an important objective of powder diffraction. Swedish workers (202) have already had much success in this area using X-ray heavy atom techniques with powders. Synchrotron radiation will mean that anomalous dispersion can also be used.

The principal advantage of the new synchrotron-sources may then not be the higher beam intensity, nor even the higher resolution possible with the extremely small natural vertical beam divergence, but the tunability of the wavelength to exploit anomalous dispersion for structure solution.

Neutron powder diffraction, with its smaller systematic

errors and absence of form factor limitations on scattering range, will probably remain superior for structure refinement.

Table 3 then represents a simplified picture of the advantages for the different radiations, summarised perhaps by the words 'structure refinement' and 'structure solution', but showing the real complementarity between high resolution neutron and synchrotron powder diffraction. Just as the high flux neutron source at Grenoble has encouraged rather than discouraged the smaller neutron scattering laboratories, the effect of the European Synchrotron Facility at a sister laboratory can be predicted by quoting Professor Guinier (203):

'a question comes naturally to mind: do the powerful Synchrotron Radiation Sources render obsolete the relatively weak old X-ray sources? We would like to show that it is not the case. On the contrary, the activity around the new sources may give an impulse to the X-ray laboratories fitted with the conventional equipment'.

TABLE 2

### Comparison of Reactor and TOF Powder Diffractometers

	Existing		New (predicted)		
	D1A/ILL	GPPD/Argonne	D2B/ILL	HRPD/SNS	
Wavelength $\lambda$	1.2–6	0.5–6	1–6	1–12	Å
Flux (sample) $\phi_s$	$2 \times 10^6$	$\sim 10^5$	$\sim 10^7$	$\sim 10^6$	$\text{n.cm}^{-1}.\text{sec}^{-1}$
Volume (sample) $V_s$	3	5	5	2	$\text{cm}^3$
Solid angle (detector) $\Omega_D$	0.004	0.1	0.008	0.1	sterad
Resolution $\Delta d/d$	2	2.5	0.5	0.5	$\times 10^{-3}$
Speed $\phi_s V_s \Omega_D$	$2.4 \times 10^4$	$5 \times 10^4$	$4 \times 10^5$	$2 \times 10^5$	

TABLE 3

### Complementarity of X-ray and Neutron HRPD

#### Advantages of X-rays

- very high resolution (espec synch)
- very fast, small samples
- high pressure and temperatures easier
- heavy atom methods
- anomalous dispersion methods (sych)

#### Structure Solution

#### Advantages of Neutrons

- high resolution — large  $d$  (long  $\lambda$ )  
— small  $d$  (no form factor)
- fast, chemical kinetics
- stress in bulk materials
- light atoms visible
- precise bond lengths

#### Structure Refinement

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# COLLEGE 6 LIQUIDS, DISORDER AND DEFECTS IN MATERIALS

## Members of the College

### I. ILL Members

J.P. Beaufils	A. Magerl
M. Bée	M. Maret
O. Chaix-Pluchery	R. Meyer
P. Chieux	R. Oberthür
D. Degenhardt	W. Petry
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T. Flottmann	A. Rennie
P. Freiländer	D. Richter
H. Godfrin	C. Ritter
R. Haensel	M. Roth
A. Heidemann	H.P. Schildberg
A. Heimig	O. Schärpf
C. Janot	W. Schirmacher
W. Just	W.G. Stirling
G. Kearley	W. Trost
W. Langel	A.F. Wright
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### II. External Members

F. Cyrot (CNRS, Grenoble)
P. Desre (ENS Electrochimie, St. Martin d'Hères)
J. Dupuy (Univ. C. Bernard, Lyon-Villeurbanne)
J.F. Jal (Univ. C. Bernard, Lyon-Villeurbanne)
F. Volino (CEN-G)
P. Becker

## GENERAL SUMMARY

The college members took advantage of the reactor shut-down period to work at other research centres in Europe and the U.S. Among those are the NBS Washington, Brookhaven National Laboratory, Argonne National Laboratory, Eidgenössische Technische Hochschule Zürich, Kernforschungsanlage Jülich and HASYLAB Hamburg. The scientific activity has been stimulated by the organisation for the 'Third International Conference on the Structure of Non-Crystalline Materials' from 8-12 July, 1985, with about 220 participants, and the workshop on 'Investigations of Higher Order Correlation Functions' (15-16 July, 1985). Both proceedings will be published by Journal de Physique C (1986).

In cooperation with the Institut für Festkörperforschung, KFA-Jülich a further workshop on 'Atomic Transport and Defects in Metals by Neutron Scattering' from 2-4 October, 1985 with about 100 participants has been held at Jülich. The proceedings will be published as a volume of the series 'Proceedings in Physics', Springer Verlag.

About half of the College 6 experiments use cold neutrons. Due to the installation of the new vertical Cold Source these experiments will profit from a flux gain of about 60%.

## SCIENTIFIC TRENDS AND HIGHLIGHTS IN 1985

### Fractons

There is now an increasing demand on experiments dealing with fractal and fracton properties. A measurement of the density of states in epoxy resins on IN6 gave first indications for a deviation of the density of state  $G(E)$  from a  $E^2$  law as it is expected for phonons (see Fig. 29). Two kinds of samples have been prepared, one hardened with the stoichiometric quantity of ethylene diamine and a second one hardened with three times the stoichiometric quantity. The second sample has shorter structural units than the first giving rise to a deviation from  $E^2$ . This behaviour coincides with what phonon-fracton theory predicts for the transition from phonon (propagating) to fracton (localized) excitations. However, theory also predicts an increase of  $G(E)$  slower than  $E^2$  for larger energies  $E$ . Up to now this has not been verified experimentally.

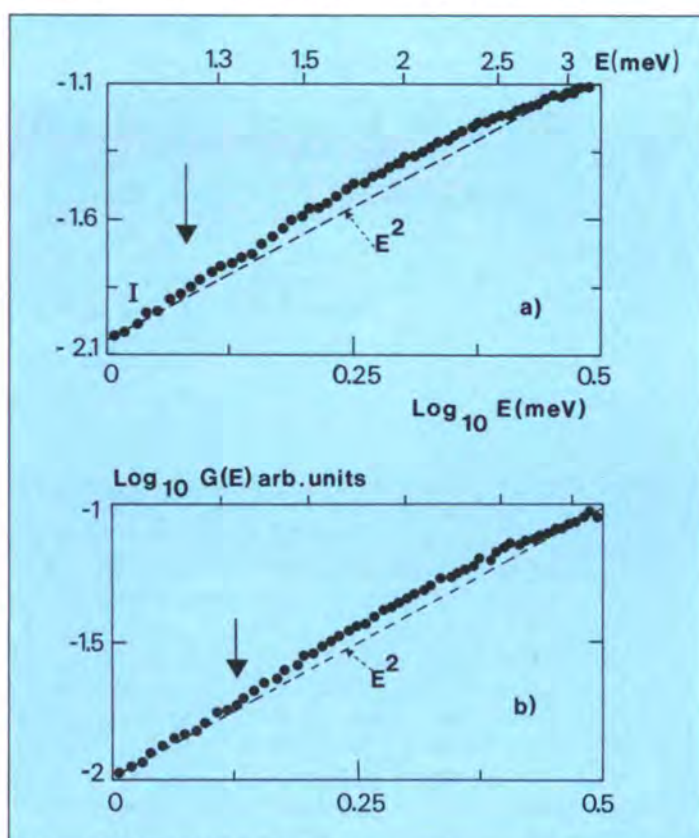


Figure 29: Log-plot of the density of state  $G(E)$  for two different epoxy resin samples measured on IN6. The arrows indicate the onset of a deviation from the  $E^2$  dependence of  $G(E)$ . Sample (a) was hardened with the stoichiometric quantity of ethylene diamine, whereas specimen (b) was hardened with three times the stoichiometric quantity.

## Quantum Liquids

There is increasing theoretical interest in the structure observed in the multiphonon continuum scattering from superfluid helium He4. It is possible that the existence of the 'extra' inelastic peaks (see Fig. 30) may help to explain outstanding problems of the roton-roton interactions in this material. A study of the roton linewidth and frequency at temperatures near  $T_\lambda$  has been initiated with much higher resolution than previously used. A damped-harmonic-oscillator function provides a much better representation of the high-temperature ( $T \approx T_\lambda$ ) lineshape than a Lorentzian function.

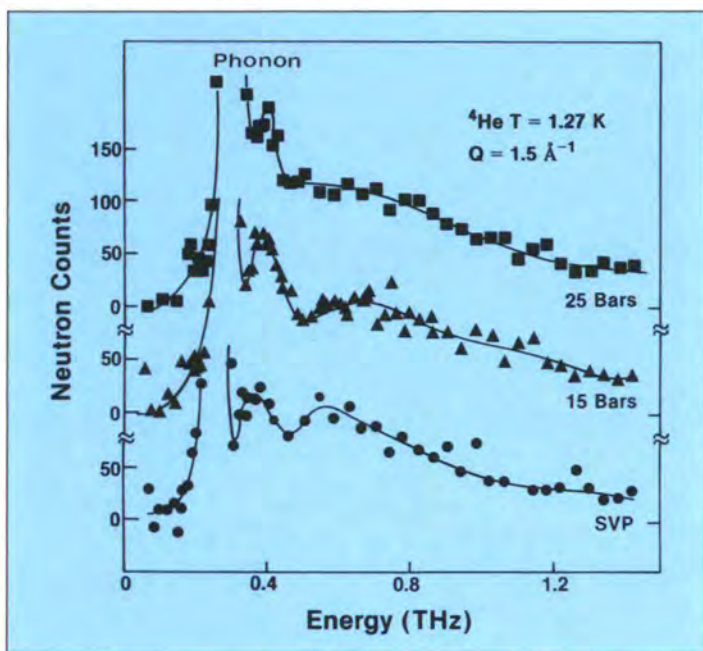


Figure 30: Multiphonon spectra of He4 showing 'structure'

## Amorphous Structures

The diffraction on the quasi-ternary amorphous alloy  $(Fe_{1-x}Mn_x)_{75}P_{15}C_{10}$  have been continued. The crucial idea of these experiments is that iron and manganese are isomorphically substituting each other. This enables, via the negative scattering length of manganese, to produce contrast variations on the iron sites and thus measure partial structure factors. Structure factors of amorphous alloys with minimum contrast between metal and metalloid site ( $x = 0.26$ ), with zero scattering length on the metal site ( $x = 0.70$ ) and a nearly zero scattering length ( $x = 84$ ) have been measured. It was found that on the average the first coordination shell contains about 13 atoms (7.26 for a non-metal and 14.82 for a metal one, mainly because of the size effects). Non-metals have been found in nearest-neighbour positions.

Additional information over distances larger than the nearest neighbours were provided from SANS experiments. The small angle diffraction pattern proved to be almost perfectly isotropic. The regrouped and corrected patterns (Fig. 31) have been fitted to a set of sphere functions which then were Fourier transformed to real space. This procedure yields a description of the inhomogeneities which are responsible for the measured SANS signals. The experimental results are consistent with a model of atoms icosahedrally packed together into 85 Å diameter 'spheres' without significant coherency losses within the measured Q range, these 85 Å diameter spheres being in

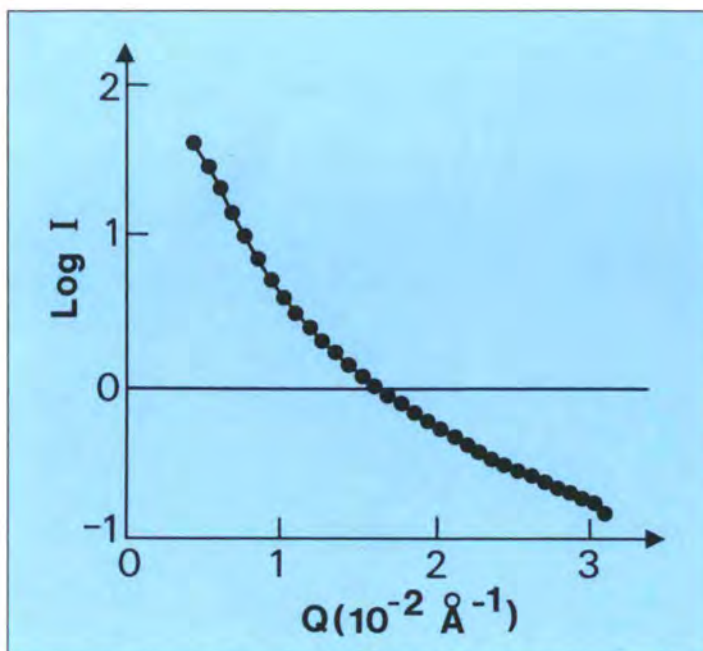


Figure 31: Regrouped low Q intensity  $I(Q)$  data of  $(Fe_{1-x}Mn_x)_{75}P_{15}C_{10}$  on a log scale.

turn packed into some kind of polytetrahedral structure. Figure 32 shows the corresponding volume distribution function  $V(d)$  with maxima at  $d_0 = 85$  Å,  $d_1 = 208$  Å,  $d_2 = 338$  Å = 1.63  $d_1$ ,  $d_3 = 440$  Å = 2.12  $d_1$ ,  $d_4 = 570$  Å = 2.73  $d_1$ ;  $d_5 = 720$  Å = 3.46  $d_1$ .

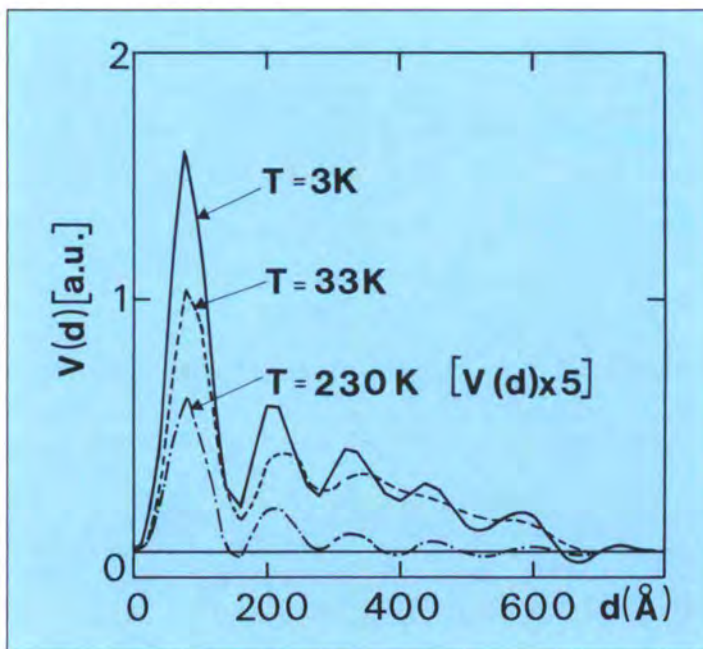


Figure 32: Distribution of the volume of the scattering particles into spheres of different diameters  $d$ , for the M26 alloys, as measured at different temperatures (the amplitudes shown are multiplied by 5 when data have been collected in the paramagnetic state).

## Diffusion in the Liquid and Amorphous Phase

Polarisation analysis measurements on liquid sodium at 840 K were performed. The improvements of the diffuse scattering machine D7 enabled the separation of the coherent and incoherent scattering at this temperature, as well as the

determination of  $S_{\text{coh}}(Q, \omega)$  and  $S_{\text{incoh}}(Q, \omega)$  by using two different wavelengths for achieving a sufficiently high resolution and extension of  $Q$ . The results were evaluated on-line. The multiple scattering and absorption correction by Monte-Carlo calculations and fits with the Lovesey model (parameter: relaxation time  $\tau(Q)$ ) and the Nelkin Ghothak model (parameter: scattering probability  $\alpha(Q)$ ) were also carried out directly on D7. The results revealed that the separated scattering functions are well described by the two models.

Motions in the fast ionic conductor  $\text{AgPO}_3 - \text{AgI}$ , which is a vitreous electrolyte, have been studied in great detail by QNS on IN6. The spectra generally consist of a quasielastic broadening of the elastic peak and of a long tail up to 10 meV which is due to an inelastic distribution. The Elastic Incoherent Structure Factor (EISF) was found to be constant at 85%. The profile of the quasielastic contribution could be well fitted by a Lorentzian line shape. When plotting the energy width  $\Delta E$  times  $S(Q)$ , the coherent structure factor, versus  $Q^2$  a straight line is obtained for all temperatures. This means that the mechanism of the conductivity is certainly a translational diffusion with a coefficient of self-diffusion of the silver ions  $D = 1.32 \times 10^{-5} \cdot \exp(-0.090 \text{ eV}/k_B T)$ . The value of the activation energy is very close to that of  $\alpha\text{-AgI}$  measured by electric conductivity. The value of EISF - 0.85 - indicates that the observed quasielastic scattering is due to half of the silver ions. These two results seem to confirm the hypothesis on the structure of these glasses: small 'clusters' of an 'amorphous' like  $\alpha\text{-AgI}$  are dispersed in the  $\text{AgPO}_3$  host glass.

## Bulk Diffusion

In the framework of diffusion studies in refractory metals (Ti) and diluted alloys ( $\text{ZrCo}$ ) a combined crystal-growing and

measuring furnace has been successfully put into operation (see photograph). It enables the growth of single crystals by the zone melting technique and keeps them hot after growth. Otherwise the martensitic phase transition hcp-bcc at above  $800^\circ\text{C}$  would destroy the single crystals when cooling down. Ti single crystals of 8 mm thickness and 40 mm long have been grown and tested in situ on the ILL  $\gamma$ -diffractometer. This furnace can be used for all kinds of elastic and inelastic neutron scattering studies on metallic single crystals which are stable at high temperatures only. A first QNS experiment has been performed on the backscattering spectrometer IN10. A Ti single crystal has been grown in situ (see photograph). Its orientation has been determined in situ on IN10 by Bragg scattering with an incident wavelength of  $\lambda = 2.33 \text{ \AA}$ . QNS measurements with the standard wavelength of IN10  $\lambda = 6.27 \text{ \AA}$  at  $1510^\circ\text{C}$  showed a pronounced variation of the linewidth with  $Q$ .

Mass transport in the non stoichiometric  $\text{Al}_{11}\text{O}_{17+x/2}\text{Na}_{1+x}$  ( $x=0.25$ ) alumina has been studied by QNS between  $-196^\circ\text{C}$  and  $400^\circ\text{C}$  on IN6. A quasielastic contribution is clearly evidenced above room temperature. It has been fitted with different jump models based on structural data. As a result, even at  $400^\circ\text{C}$  only 15% Na cations are mobile on the time scale of IN6 ( $\tau < 10^{-10}\text{s}$ ). Two kinds of motions are characterised: localised jumps ( $\tau < \sim 10^{-12}\text{s}$ , activation energy 0.050 eV) and long range diffusion ( $D \sim 0.75 \cdot 10^{-5} \text{ cm}^2 \text{ s}^{-1}$ , activation energy 0.25 eV).

## Dynamics of Hydrogen in Crystalline and Amorphous Materials

In the field of hydrogen motions in solids there is a tendency to move away from simple host metals to more complicated

*The Third International Conference on the Structure of Non-crystalline Materials (8-12 July 1985) with more than 220 participants and organized by the ILL was a great success.*





View of the furnace combining crystal growth in situ and neutron scattering experiments.

systems like amorphous metals or systems with multiple site occupation.

As an example of multiple site occupation the motion of hydrogen in  $\beta$ -LaNi<sub>5</sub> and  $\beta$ -LaNi<sub>4.5</sub>Al<sub>0.5</sub> has been studied with different energy resolutions. The QNS spectra were decomposed into at least three components: one representing the elastic intensity, and two broadened components of Lorentzian line shape. The results were successfully interpreted by assuming the existence of two types of long range (diffusive) motion in addition to a rapid localized motion. The latter gives rise to a broader quasielastic component. These types of motion are related to the availability of several interstitial sites for hydrogen as they are known from earlier diffraction studies. It is assumed that hydrogen diffuses easily between the 6m, 4h and 12o sites near the  $z = 1/2$  plane, whereas the diffusion from 3f and 12n sites near the basal plane is much slower. These latter sites form a cluster in which the hydrogen atoms perform rapid localized jumps. The substitution of Ni by Al does not affect the localised motion but slows down the diffusive motion.

Using the time of flight technique quasielastic and inelastic neutron scattering have been combined to reveal some aspects of the dynamics of hydrogen in the amorphous alloy CuTi. From the quasielastic broadening of the neutron spectra it has been concluded that part of the hydrogen atoms perform a fast localised motion occurring on a short length scale. The activation energy of this motion is around 0.3 meV. The high

frequency mode around 140 meV reveals the preferential filling of Ti<sub>4</sub> tetrahedral at low concentration of hydrogen. The temperature dependence of this mode indicates the formation of more Ti<sub>4</sub> sites as the temperature is increased. The metal vibrational mode around 20 meV is enhanced by the introduction of hydrogen, giving a measure of the hydrogen-metal interaction in these amorphous hydrides.

By means of QNS the long range diffusion of hydrogen in amorphous Pd<sub>1-x</sub>Si<sub>x</sub>H<sub>y</sub> ( $x = 0.20$  and  $0.15$ ) was studied. This investigation led to the surprising discovery of two well separated regimes of slow and fast hydrogen jump rates. Figure 33 presents the quasielastic broadening as a function of the momentum transfer  $Q$  as obtained from Lorentzian fits to the experimental spectra. The narrow line was measured on IN10 while the broad component was obtained on IN5.

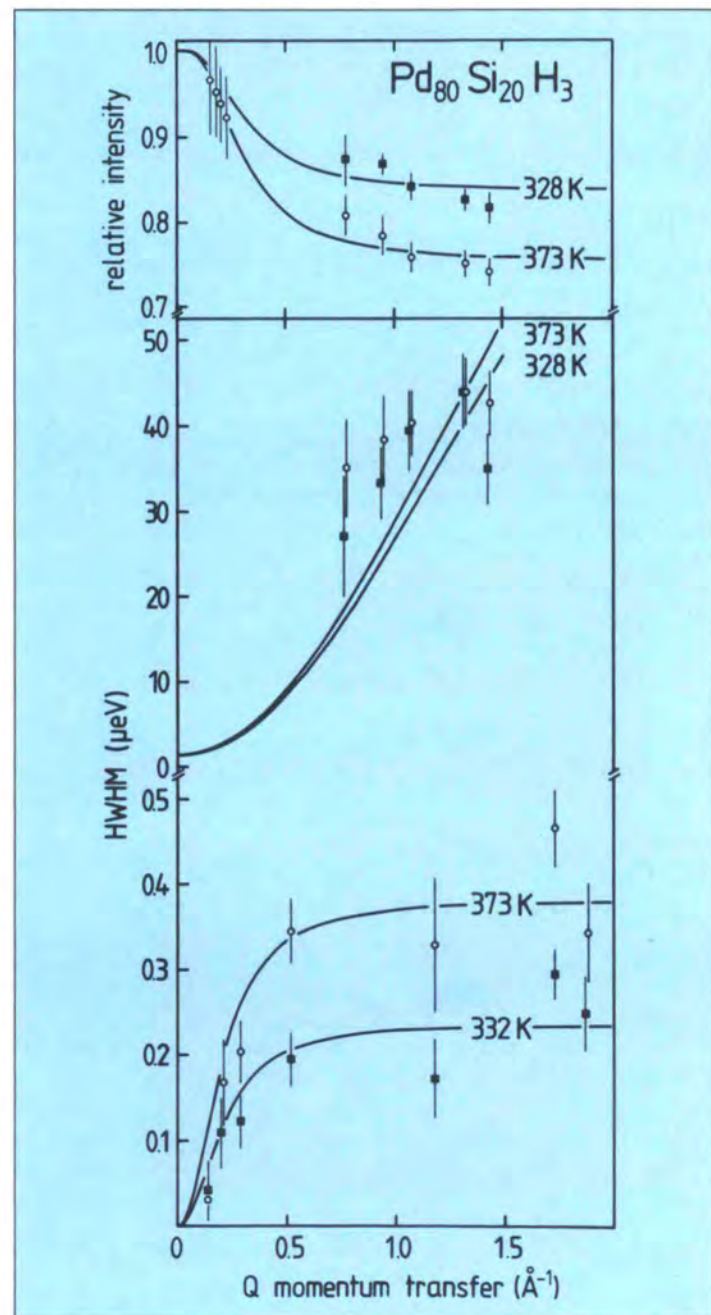


Figure 33: Upper part: weight of the elastic component in the IN5 spectrum as a function of temperature and momentum transfer. Lower part:  $T$  and  $Q$  dependence of the quasielastic linewidths (HWHM) observed on IN5 and IN10. The solid line represents a fit of the two state model to these data.

Both linewidths differ by about two orders of magnitude. This observation is in clear contradiction to previous ideas on H transport in amorphous metals which assumed a continuous Gaussian like distribution for the H site energies and thus the H jump rates. In the upper part of Fig. 33 the relative weight of the narrow component in the time of flight spectra is shown. The weight of the fast component vanishes at small Q indicating the spatial restriction of the corresponding jump process. With increasing temperature the fast jumps gain in weight; apparently the mobile state is reached by thermal activation. A consistent interpretation of these features is possible in terms of a two state diffusion and trapping model, where the slow jumps correspond to escape processes out of energetically favourable sites or traps while the fast jumps are connected with the diffusion in between such traps. The single narrow Lorentzian at small Q reflects the long range effective diffusion including trapping and release processes. The solid lines in Fig. 33 are the results of a fit with a two state model. Microscopically the experimental results can be taken as evidence for the existence of two different kinds of H-interstices, possibly octahedral and tetrahedral sites. Those with the lower site energy act as traps for the diffusing proton.

## Spinodal Decomposition

Macroscopic density fluctuations of the interstitially dissolved hydrogen in niobium near the gas-liquid like phase boundary have been observed in a very direct way via neutron radiography. A gradient over the whole sample ( $\sim 2$  cm) builds up which gives rise to regions of different hydrogen concentrations of about  $\Delta c = 0.15$  [H/Nb]. The theory of spinodal decomposition predicts this kind of mode, if the *elastic interaction* between the hydrogen is taken into account. This thermodynamic effect concerning the whole sample and being shape-dependent is a striking evidence for the predominant rôle of the elastic interaction in  $\text{NbH}_x$  at the critical point (Fig. 34).

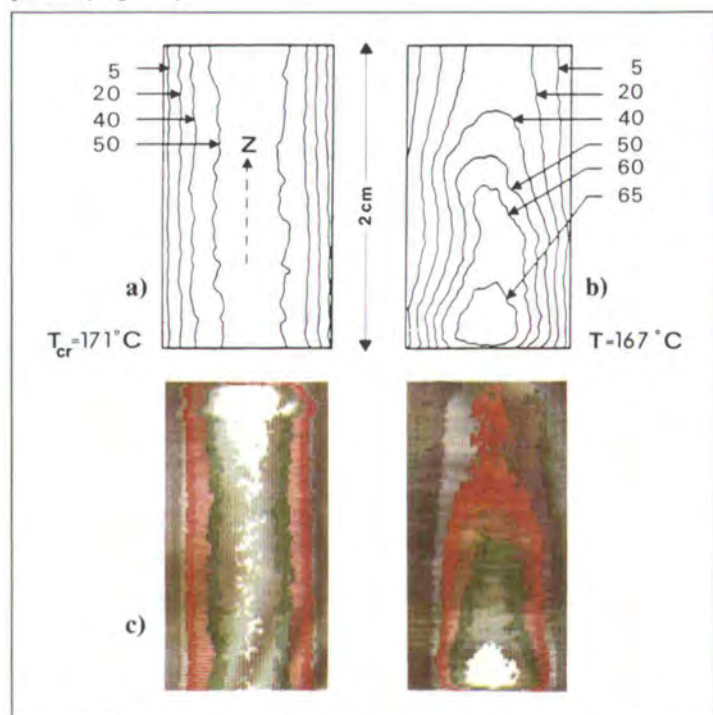


Figure 34: Absorption levels (arbitrary units) before and after spinodal decomposition in Nb of cylindrical shape. (a) homogeneous H-distribution at the critical point  $T_{cr} = 171^\circ\text{C}$ ; (b) inhomogeneous H-distribution at  $4^\circ$  below the critical point; (c) same as (a) and (b) in a colour plot.

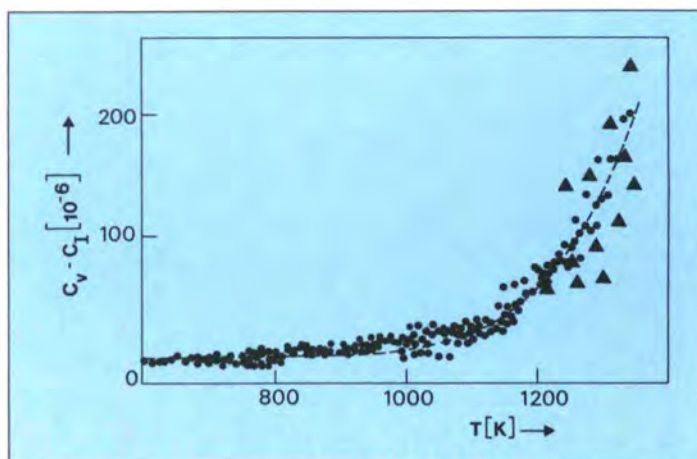
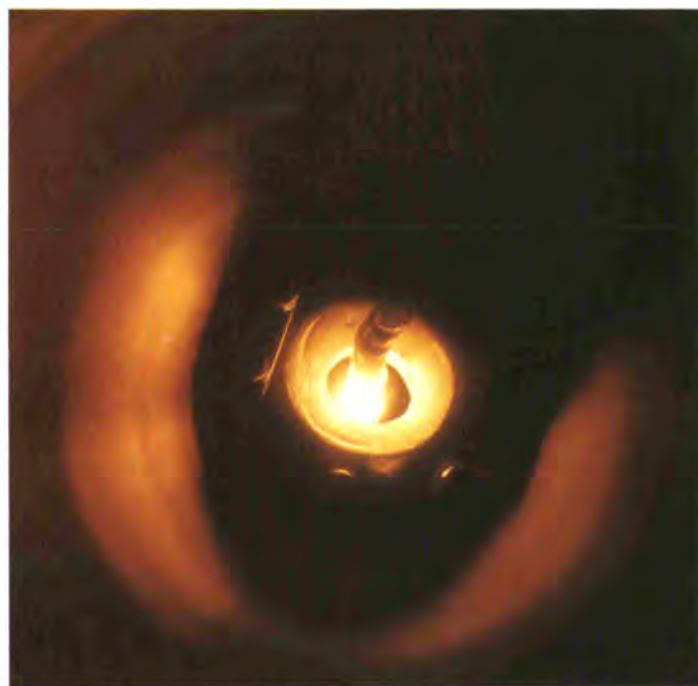


Figure 35: Concentration difference  $C_V - C_I$  in Cu as a function of temperature. The dashed line corresponds to the fit  $c = \exp(S/k_B - H/k_B T)$  with  $S = 1.6 k_B$  and  $H = 1.18 \text{ eV}$ . Data measured by Simmons and Balluffi (Phys. Rev. 1963) are shown by triangles.



Growing of a Ti single crystal by zone melting. Temperatures in the molten zone are between  $1660^\circ\text{C}$  and  $1700^\circ\text{C}$ . During the growth of the crystal, the furnace is mounted on the IN10 backscattering spectrometer.

## Point Defects in Metals in Thermal Equilibrium

The only way to measure point defect concentrations in thermal equilibrium on an absolute scale is to measure simultaneously the macroscopic expansion  $\Delta l/l_0$  and the microscopic lattice parameter change  $\Delta a/a_0$  (so-called Simmons-Balluffi experiment). This method allows correction for the 'defect free' thermal expansion which is not known. High precision is required since the desired information is obtained as a small difference of two strongly temperature-dependent quantities. In recent years a laser interferometer (to measure  $\Delta l/l_0$ ) was built up on the double crystal diffractometer S21 (to measure  $\Delta a/a_0$ ). Measurements on high quality Cu and Au single crystals showed that a precision of  $2 \times 10^{-6}$  in  $\Delta l/l_0 - \Delta a/a_0$  has been reached. This is about one order of magnitude better than the accuracy achieved by the original work of Simmons and Balluffi.

Fits to the measured defect concentration (difference between vacancy and interstitial concentration  $c_v - c_i = 3(\Delta\ell/\ell_0 - \Delta a/a_0) = \exp(S_{\text{eff}}/k_B) \cdot \exp(H_{\text{eff}}/k_B T)$  yields an effective entropy  $S_{\text{eff}} = 1.6 k_B$  and an effective enthalpy  $H_{\text{eff}} = 1.18 \text{ eV}$ . At the melting point one finds  $c_v(T_m) - c_i(T_m) = 2.1(\pm 0.1) \cdot 10^{-4}$  in excellent accordance with the Simmons-Balluffi value of

$1.9(\pm 0.5) \cdot 10^{-4}$  (see Fig. 35). However, the question whether self interstitials do or do not contribute substantially to the equilibrium concentration of point defects in metals has still to be considered as open.

Secretary: W. Petry

### ICOSAHEDRAL SYMMETRY AND QUASICRYSTAL DIFFRACTION

At the beginning of last year, were reported the first observations of 5-fold rotational symmetry in the structure of solid specimens of a manganese-aluminium alloy which in composition approximates  $\text{Al}_6\text{Mn}$  and is obtained by fast-quenching from the melt.

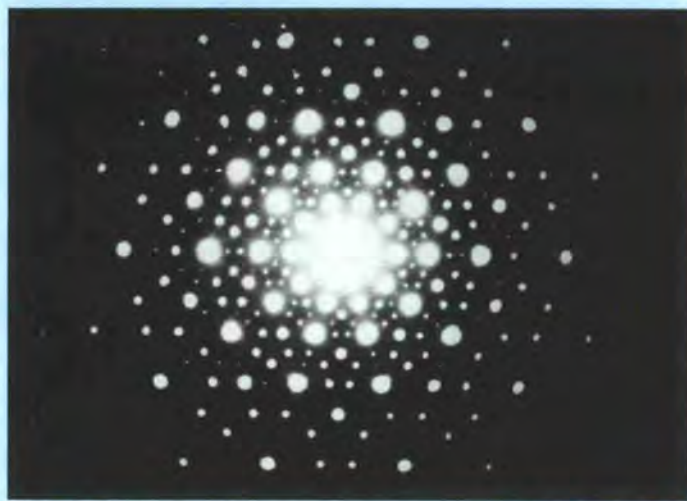


Figure 36: Electron diffraction pattern of a  $\text{Al}_6\text{Mn}$  quasi-crystal alloy showing 5-fold symmetry.

Electron diffraction (see Fig. 36) in the appropriate direction yields patterns of sharp spots with obvious 5-fold symmetry. According to the elementary textbooks five-fold symmetry axes are not consistent with the translational symmetry of a crystal lattice. For instance it is not possible to cover the two-dimensional plane with regular pentagons (Fig. 37). However the difficulty can be bypassed if a so-called Penrose tiling is used. The Penrose covering of the two dimensional plane (see one example if Fig. 38) associates tiles shaped like one or other of two distinct rhombi. Such a tiling obviously has some strict 5-fold axes of rotation perpendicular to the plane and the boundaries between tiles have orientations belonging to a finite set of directions in the plane. It is also very clear looking at Fig. 38 that the price paid for pentagonal symmetry, is that strict translational order is lost.

Three-dimensional generalizations of a Penrose coverings can be built to explain 5-fold symmetry diffraction pattern. In these framework of atoms, neighbour-neighbour bonds have fixed orientation but translational periodicity is replaced by 'quasi-periodicity': all points are given by integral combinations of vectors with lengths incommensurate with each other. Then, quite naturally, has emerged the notion of quasi-crystalline matter.

However the icosahedral phase of  $\text{Al}_6\text{Mn}$  still provides a rare challenge to crystallographers. The incomplete state of knowledge can be appreciated by considering the fact that even for the most complicated cases of ordinary crystal struc-

ture at least the determination of the lattice parameters is a straightforward task. The Al-Mn icosahedral phase defies even this first step of the structure determination. There is so far no indication that in either real or reciprocal space its structure can be described by simply specifying the dimensions and angle of some periodically repeating unit.

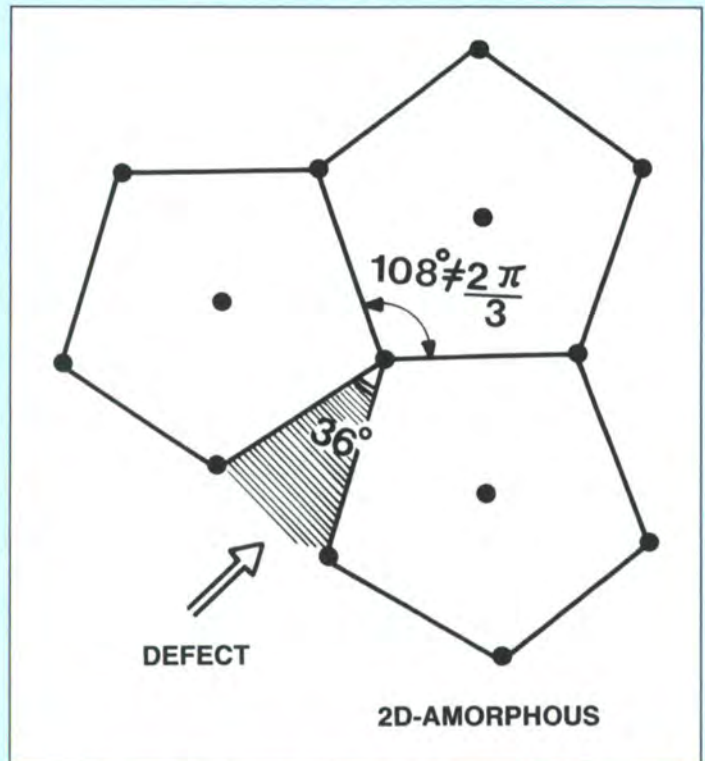


Figure 37: 2D-space filling is not possible with regular pentagons.

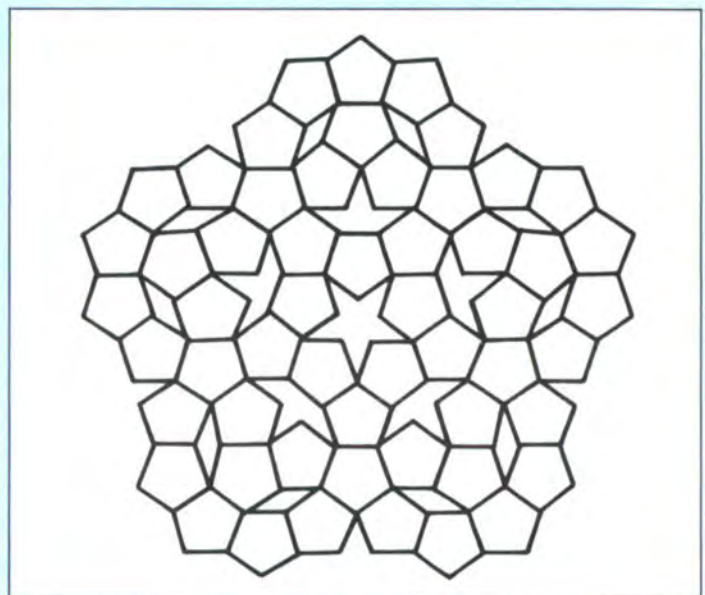


Figure 38: Example of a Penrose tiling.

# COLLEGE 8 AND EMBL GRENOBLE BIOCHEMISTRY

## Members of the College

### I. At ILL

L.F. Braganza  
A.J. Dianoux  
K. Ibel  
M.S. Lehmann  
S. Mason  
R. May  
G. McIntyre  
R. Oberthür  
A. Rennie  
M. Roth  
P. Timmins  
J. Torbet  
S. Wilson  
G. Zaccai

### II. At EMBL

A. Bentley  
G.A. Bentley  
C. Berthet  
J. Chroboczek  
M. Cuillel  
S. Cusack  
C. Devaux  
A. Gabriel  
F. Guinet  
M. Härtlein  
B. Jacrot  
J.C. Jesior  
J. Langowski  
R. Leberman  
K. Weckström  
D. Wild  
M. Zulauf

## INTRODUCTION

During the long reactor shutdown in 1985, experiments were necessarily delayed and most College members took the opportunity to strengthen existing collaborations and generate new ones by visits to other laboratories.

The small-angle diffractometer D11 has been rebuilt and the detector which is now mounted on a trolley can be readily placed at any sample to detector distance between 1 m and 37 m; access to the sample area is also easier than before. These major improvements have made D11 easier to use and will allow greater flexibility during experiments. The jointly financed ILL/EMBL four circle diffractometer DB21 with a two-dimensional PSD for low resolution crystallography of biological macromolecules is currently being tested and expected to be operational by February 1986.

In April 1985, the ILL/EMBL acquired a PS300 Evans and Sutherland colour vector graphics display system which has already proven to be an invaluable tool for the display and manipulation of diffraction data from proteins, viruses and small molecules. This is particularly so in the case of matrix porin and satellite tobacco necrosis virus (STNV) where low resolution structures have been obtained and for tomato bushy stunt virus (TBSV) where the structure is currently being refined.

## SCIENTIFIC TRENDS AND HIGHLIGHTS IN 1985

A meeting of the French Biophysical Society on 'Complementary Physical Approaches in Structural Biology' was held in Autrans near Grenoble in October 1985. Sponsors included the ILL and EMBL and the organisation carried out jointly by ILL/EMBL. Of the 100 participants, the ILL/EMBL was strongly represented with 10 ILL/EMBL members attending, most of these as speakers. The conference included topics on neutron and X-ray diffraction, NMR, and electron diffraction and microscopy.

### Structure and Interactions in Solution

Solvent environment is important for biological function and small angle neutron scattering (SANS) from solution is now well established as a means of studying the influence of solvent on protein and nucleic acid structures and protein-nucleic acid interactions. In the past year, a number of studies in this field have been completed including the structural characterisation of transfer RNA in a broad range of different salts (ILL). Studies of the ternary complex formed between bacterial elongation factor Tu, GTP and valyl tRNA<sup>val</sup>, show that the protein in absence of tRNA is in a monomer-dimer equilibrium which is shifted towards the monomer as the complex is formed. The complex has a compact structure in the solvent conditions examined (EMBL, ILL). The behaviour of this system is similar to that of certain amino acid tRNA synthetases where the protein also interacts specifically with transfer RNA (Strasbourg, Palaiseau, ILL). One of these is the homologous dimer methionyl-tRNA synthetase, where using selective deuteration, the structure of protomers within the dimer has been studied (Palaiseau, ILL). The methods used to study tRNA-protein interactions are now being applied to the study of DNA-protein interactions, for instance in the DNA-gyrase complex.

The solvent associated with halophilic proteins has also been studied. Following neutron and X-ray experiments a structure



Figure 39: A model for halophilic malate dehydrogenase in high salt. The core is similar to the crystal structure of non-halophilic malate dehydrogenase and the loops are in strong interaction with water and salt from the solvent.

has been proposed for halophilic malate dehydrogenase which is an enzyme that functions only under high salt conditions. The model is that of a compact protein core, the catalytic moiety stabilized by loops extending into the solvent. This protein has much more solvent associated with its structure than non-halophilic proteins (ILL, Weizmann Institute) (see Fig. 39).

### E. Coli Ribosomal Subunit

A refined treatment of the complete data set obtained so far has led to a preliminary model of the positions of 11 of the 32 proteins within the large ribosomal subunit of E. Coli. This model is based on a restricted number of 31 inter-protein distances. The handedness has been chosen using biochemical information and the results of electron microscopy. It is expected that certain protein positions will change as further data is incorporated into the model. This is especially true for protein L13 the arrangement of which has been selected arbitrarily from two possible mirror positions. A more complete model will result after data obtained in November 1985 has been included (single proteins L4 and L16, and protein distances L2-L20, L2-L21, L9-L16, L9-L22, L13-L21, L15-L16 and L21-L22) (see Fig. 40).

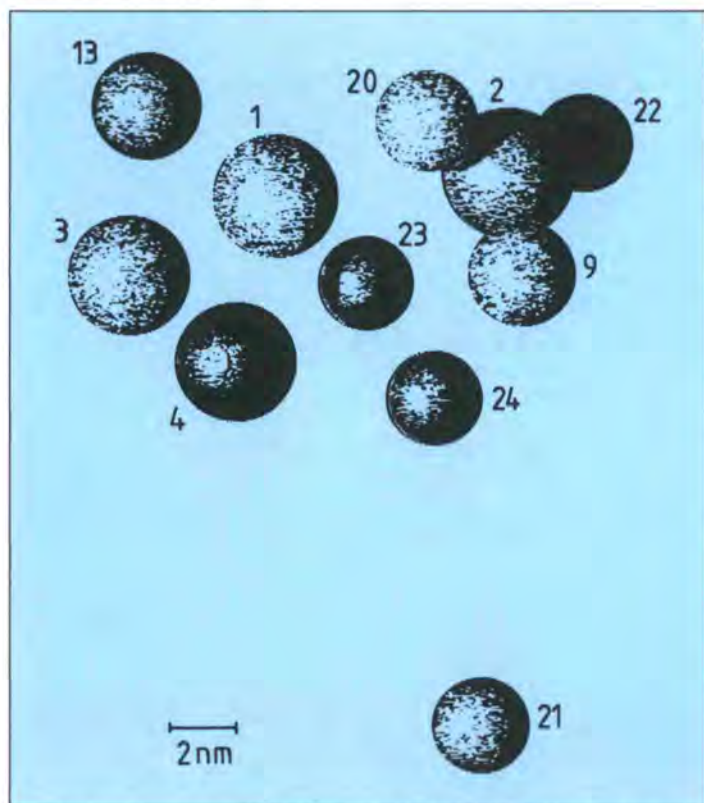


Figure 40: The positions of the eleven proteins of 50S ribosomal subunit from *Escherichia coli* are arbitrarily depicted as spheres the radii of which correspond to their molecular masses. The spheres are shaded in order to show the 3-dimensional arrangement of proteins.

### Low Resolution Crystallography

Matrix porin is a trimeric transmembrane protein found in the outer membrane of E. Coli and functions as a voltage gated pore which allows the passage of small molecules of molecular weight  $\ll 700$ . Once the protein has been extracted from its membrane environment using the detergent octyl glucoside, matrix porin can be readily crystallized. Crystals are produc-

ed with two trimers per asymmetric unit which diffract X-rays to 2.8 Å resolution. It has not, however, been possible to produce isomorphous derivatives which are essential for X-ray structure determination and so a neutron diffraction study has been carried out on single crystals using H<sub>2</sub>O/D<sub>2</sub>O solvent contrast variation. In H<sub>2</sub>O, the detergent is almost contrast matched out, and applying model fitting and molecular replacement methods to the data, a 16 Å resolution structure of the protein has been obtained. The current model shows that the three distinct pores seen at the external membrane surface do not merge into a single channel. However, it is not possible to categorically exclude the model obtained from electron microscopy studies, which suggests that the three pores meet to form a single channel. The phases obtained for the structure factors in H<sub>2</sub>O are being extended to other contrasts where the detergent scattering predominates in order to obtain the arrangement of detergent molecules around the protein (ILL, EMBL, Basel) (see Fig. 41).

Contrast variation studies on single crystals of tomato bushy stunt virus (TBSV) have been able to locate the RNA and the 25% of the total protein present which cannot be visualized in electron density maps from X-ray diffraction. Preliminary results show the RNA to be located in a narrow shell below the protein coat, and the protein not seen in X-ray studies to be found inside this radius. The structure is currently being refined using the non-crystallographic 5-fold symmetry of the viral particle (ILL, EMBL, Strasbourg) (see Fig. 42).

Satellite Tobacco Necrosis Virus (STNV) is a simple icosahedral plant virus composed of a single strand of RNA (1239 nucleotides) enclosed by a protective coat of 60 identical protein subunits. The crystal structure of STNV has been studied by neutron diffraction at low resolution ( $d_{\min} = 16$  Å) in order to locate the RNA which is not visible in the 2.5 Å resolution X-ray analysis. This was achieved using H<sub>2</sub>O/D<sub>2</sub>O solvent contrast variation to match out alternately the low resolution diffraction from the protein and the RNA. Diffraction data was collected from four crystals of STNV which had been soaked in buffers of different H<sub>2</sub>O/D<sub>2</sub>O ratios. The structure of the RNA was determined using the knowledge of the protein structure from the X-ray analysis and the non-crystallographic symmetry of the virus. The RNA was found to be largely confined to the regions just inside the external protein coat of the virus. It is made up of two principal structural motifs: (a) a rod of density in intimate contact with the helical N-termini of the protein subunits, and (b) a sphere of density located on each of the icosahedral 5-fold axes at about 65 Å from the centre of the virus. These two motifs of density are repeated by the icosahedral symmetry elements to cover the inside surface of the protein coat (ILL, EMBL) (see Fig. 43a and b).

Secretary: L.F. Braganza  
Head of EMBL Outstation: B. Jacrot

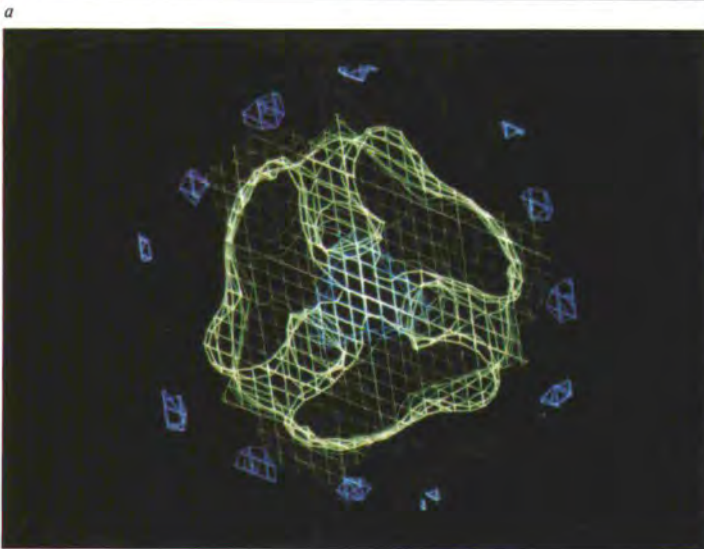
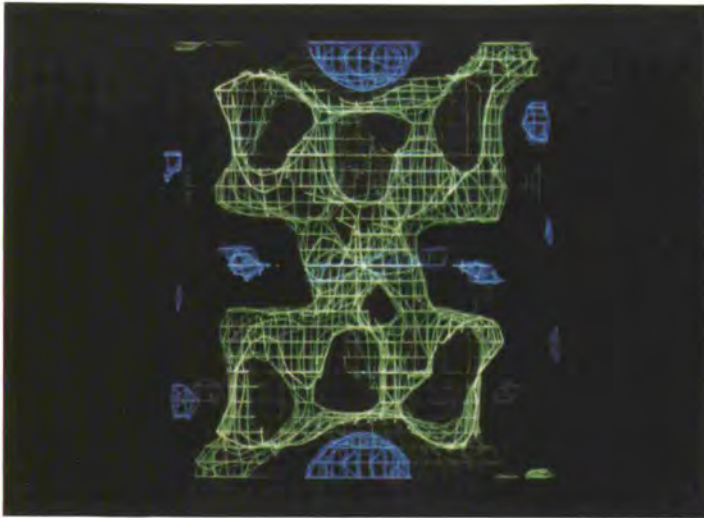


Figure 43a: The RNA density in STNV is mainly located just inside the external protein coat. It is shown here in relation to five protein subunits which are related by a 5-fold rotation axis.

Figure 41: Scattering length density maps of matrix porin in 0%  $H_2O$ .  
(a) Map showing a dimer of trimers (the crystallographic asymmetric unit). Important features of the model include the three pores penetrating the trimer and the 'feet' forming the interface between trimers.  
(b) View down the 3-fold axis of the porin trimer showing the pores and suggesting the sub-unit boundaries.

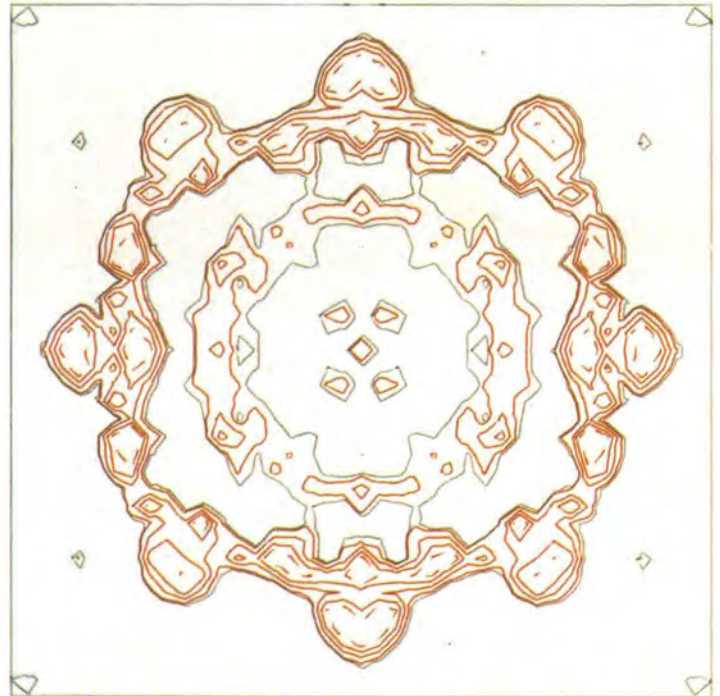


Figure 42: Scattering length density map from TBSV virus in 70%  $D_2O/H_2O$  - section through the centre of the virion. At this contrast the RNA is essentially invisible and the map shows the protein coat (as determined by X-ray diffraction) and an internal shell in which is located the protein not identifiable by X-ray studies.

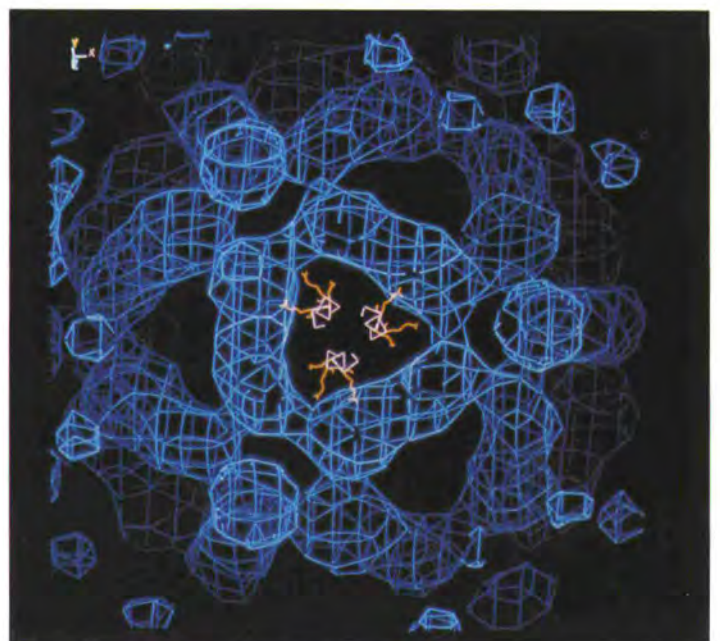


Figure 43b: A view of the RNA density down an icosahedral 3-fold axis showing its close interaction with the helical N-termini of the protein subunits. The protein side chains shown are the positively charged lysine and arginine residues which interact (presumably) with the negatively charged phosphate groups at the RNA.

# COLLEGE 9 CHEMISTRY

## Members of the College

P. Chenevas  
R. Ghosh

M. Lehmann  
P. Pautrot

## 9a: Molecular Spectroscopy, Surfaces and Mesophases

J.P. Beaufils  
M. Béc  
H. Blank  
A.J. Dianoux  
A. Fitch  
A. Heidemann  
G. Kearley  
H. Godfrin  
W. Langel

H.J. Lauter  
A. Magerl  
J. Pannetier  
W. Petry  
O. Chaix-Pluchery  
C. Poinsignon  
C. Ritter  
B. Saubat-Marcus

## 9b: Colloids and Polymers

P. Chieux  
B. Farago  
K. Ibel  
J. Kugler  
P. Lindner

R. Oberthür  
M. Rawiso  
A.R. Rennie  
D. Richter  
A.F. Wright

## INTRODUCTION

As with other Colleges, the scientific work was greatly affected by the long shut-down of the reactor. Many physicists spent periods visiting other laboratories. The experimental programme resumed in September and the new cold source produced flux gains on many instruments of interest to the College. The rebuilt small-angle scattering instrument D11 is already proving popular with users; the possibility of rapid changes in detector position has permitted more investigative experiments.

## SCIENTIFIC HIGHLIGHTS IN 1985

### Rotational and Translational Motions

Quasielastic scattering is now frequently used to investigate the motions in more complex molecules and environments. In the case of the perovskite-type layer compounds  $(\text{NH}_3(\text{CH}_2)_3\text{NH}_3)\text{MnCl}_4$  deuteration of the  $\text{CH}_2$  units and separately of the  $\text{NH}_3$  groups enabled the QNS spectra to be fitted successfully in terms of  $\text{NH}_3$  reorientations and motions of the  $(\text{CH}_2)_3$  backbone. As the temperature is increased in the ordered phase, there is a pronounced softening of the  $\text{NH}_3$  torsional mode as the phase transformation is approached. Similar behaviour has been observed for the torsional modes of the  $\text{NH}_4^+$  ion in  $\text{NH}_4\text{PF}_6$ , as the III II transformation is approached (Fig 45) (ILL).

Rotational dynamics in systems such as liquid cyclopentane and hexaoxacyclo-octane are rather difficult to study by QNS due to the poor sensitivity to the anisotropy of the angular

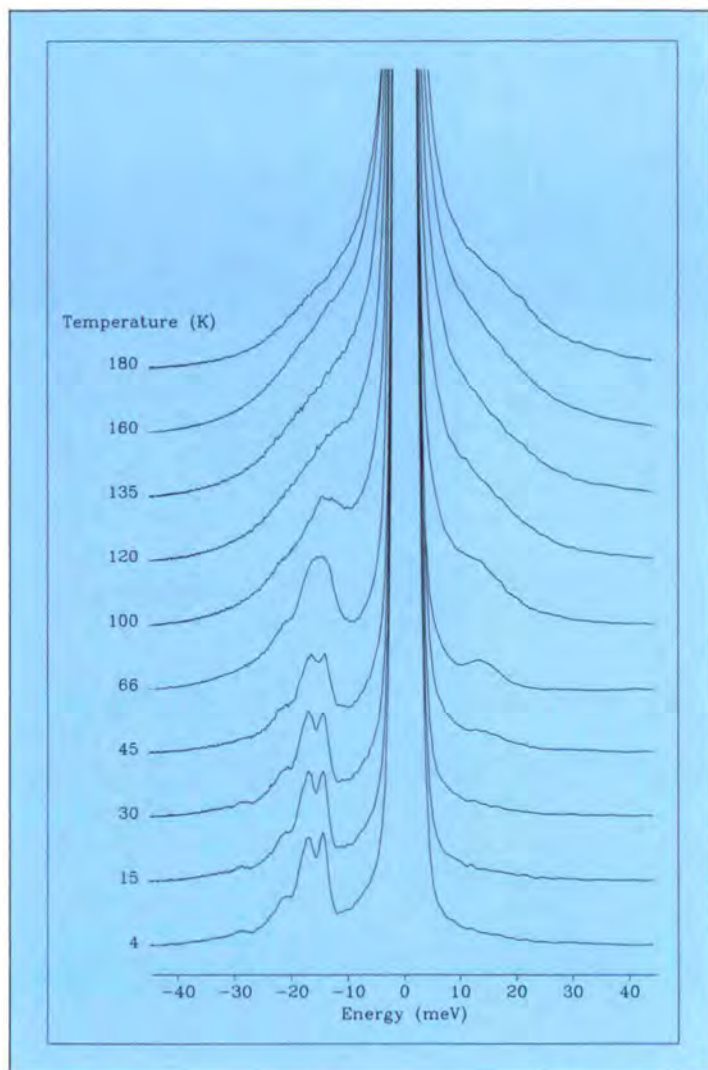


Figure 45: Temperature dependence of the torsional modes of  $\text{NH}_4^+$  in  $\text{NH}_4\text{PF}_6$ .

motions. The conformational dynamics however, have been successfully studied (ILL, CNRS) since these motions occur on a much shorter time-scale than the whole molecule diffusion.

The dynamical behaviour of supercooled water in porous oxide gels has been followed by variation of the water content from one absorbed water layer through to the sol state (30% water) (Harwell, ILL). Low frequency vibrational modes of the water molecules have also been used to examine gels with different porosity.

QNS has been used to study the rotational and translational motions of  $\text{HNO}_3$  intercalated into graphite. Whilst at temperatures below 210K a commensurate structure exists, between 210 and 260K a semi-commensurate structure appears in which only rotational motions are found. The number of rotating molecules in this phase is temperature dependent. In the liquid phase ( $>260\text{K}$ ) translational and rotational motions occur within the two-dimensional environment.

### Molecular Vibrations

In an attempt to improve the analysis of molecular vibrations with neutrons, a method has been developed in which interatomic force-constants are refined directly to the observed spectral profile (ILL). This approach is the most effective way

of including frequency and intensity data into the vibrational analysis. Clearly, the significance of the refined parameters rests heavily on the data quality and accuracy of the scattering function. Nevertheless, reasonable agreement between observed and calculated spectra can be obtained even using a fairly approximate scattering function (Fig 46). More recently, this treatment has been extended to include multi-phonon modes and the use of pulsed- source spectrometers (R.A.L., ILL).

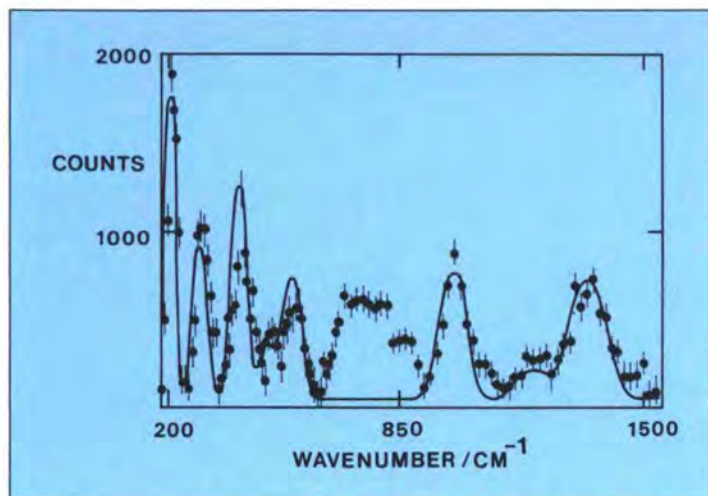


Figure 46: Observed and calculated vibrational spectra of  $\text{CH}_3\text{CCO}_3(\text{CO})_9$ . The region between  $600$  and  $900\text{ cm}^{-1}$  is excluded from the analysis.

## Matrix Isolation Spectroscopy

Neutron scattering allows various aspects of molecules isolated in rare-gas matrices to be studied. In the thermal energy-range, the vibrational modes of the trapped molecules and the perturbation of the phonon density of states of the matrix have been observed (Siegen, ILL). The observation of small molecular aggregates which are separated from each other is of special interest since the internal density of states of these clusters is markedly different from that of the corresponding pure phases.

At much lower energies, tunnelling spectra of matrix isolated species have been observed for the first time using neutron scattering. A comparison of the tunnelling spectrum of  $\text{Sn}(\text{CH}_3)_4$  in argon with that of pure  $\text{Sn}(\text{CH}_3)_4$  allows a separation of intra and intermolecular potentials by experimental means.

## Kinetic Studies

Analysis of the structure and dynamics of  $\text{ABF}_6$  salts are now mostly complete for  $\text{A} = \text{K}^+$ ,  $\text{NH}_4^+$  and  $\text{B} = \text{P}$ . Investigations are now progressing towards other cations (Na, Cs, Rb) and embracing kinetic studies. For example, D1B has been used to investigate the phase transition from rhombohedral (ordered CsCl-type structure) to cubic (disordered NaCl-type structure) in the case of  $\text{KPF}_6$  (Fig 47). A hydrostatic pressure-jump method was used, the equivalent temperature studies being impossible due to the large thermal capacity of powdered samples. This approach enables rate-constants for the transition to be obtained and hence the thermodynamic quantities of activation energy and volume may be determined. The results help to explain the observation that freshly precipitated

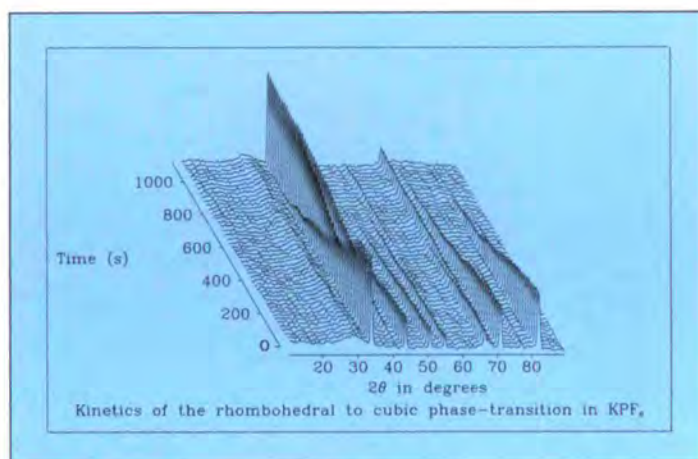


Figure 47: Kinetics of the rhombohedral to cubic phase transition in  $\text{KPF}_6$ .

$\text{KPF}_6$  transforms to a rhombohedral phase on cooling whilst dried thermally cycled  $\text{KPF}_6$  transforms to a monoclinically distorted NaCl-type modification under the same conditions.

## Physisorption

Physisorbed systems can be investigated by various methods. A short outline of the application of X-ray diffraction is given under the contribution of College 4.

Much interest is given now to neutron diffraction studies of physisorbed layers on MgO-powder.  $\text{CD}_4$  forms a  $2 \times 2$  overstructure on MgO (Marseille, Paris, ILL). The adsorbed monolayer melts directly out of this registered phase. This melting process is studied in addition by the investigation of the quasielastic line-width. The commensurability of the solid phase extends at least up to four adsorbed layers verified by a measured structure factor which varies as a function of coverage. Also  $^{36}\text{Ar}$  on MgO is commensurate with the substrate and the data analysis is in progress (ILL, Marseille).

The investigation of physisorbed systems on graphite concentrated on the transition between the  $(\sqrt{3} \times \sqrt{3})$  structure and the more dense incommensurate phase of quantum systems ( $\text{He}$ ,  $\text{D}_2$ ). These systems are governed mainly by the repulsive interaction between the adsorbed atoms. The appearance of satellite peaks indicates the creation of domain walls in this region ( $\text{D}_2$  on graphite) (Mainz, ILL). Also, the study of two adsorbed layers of  $^4\text{He}$  and  $\text{D}_2$  on graphite continued (ILL, Mainz). The interaction of the layers manifests itself, when the melting of the second layer on top of the solid first layer was studied.

Finally, the wetting transition of multilayers of  $\text{CF}_4$  and  $\text{O}_2$  on graphite will be studied soon (Marseille, ILL).

## Colloids and Emulsions

Experiments on the physical chemistry of colloids have continued to involve a wide variety of different systems ranging from inorganic particles, through polymeric lattices to microemulsions. The study of concentrated systems, strongly interacting particles and adsorbed surface layers have attracted particular attention.

Colloids that crystallize allow effects that are observed normally only on atomic or molecular scale to be studied with larger particles. It is thus possible to observe the result of varying for example concentration or interaction parameters on

melting and other transitions. A recent experiment on D17 measured the single crystal diffraction pattern of an oil/water micro-emulsion (Saclay).

The fluid properties of colloids were emphasised in another experiment (Bristol/ILL collaboration) on the rebuilt D11 small angle scattering instrument. A concentrated polymethyl methacrylate/dodecane colloid was subjected to shear at rates up to  $10000 \text{ s}^{-1}$ . The changes in the peak position corresponding to the inter-particle spacing were observed as a function of shear rate (see Fig 48).

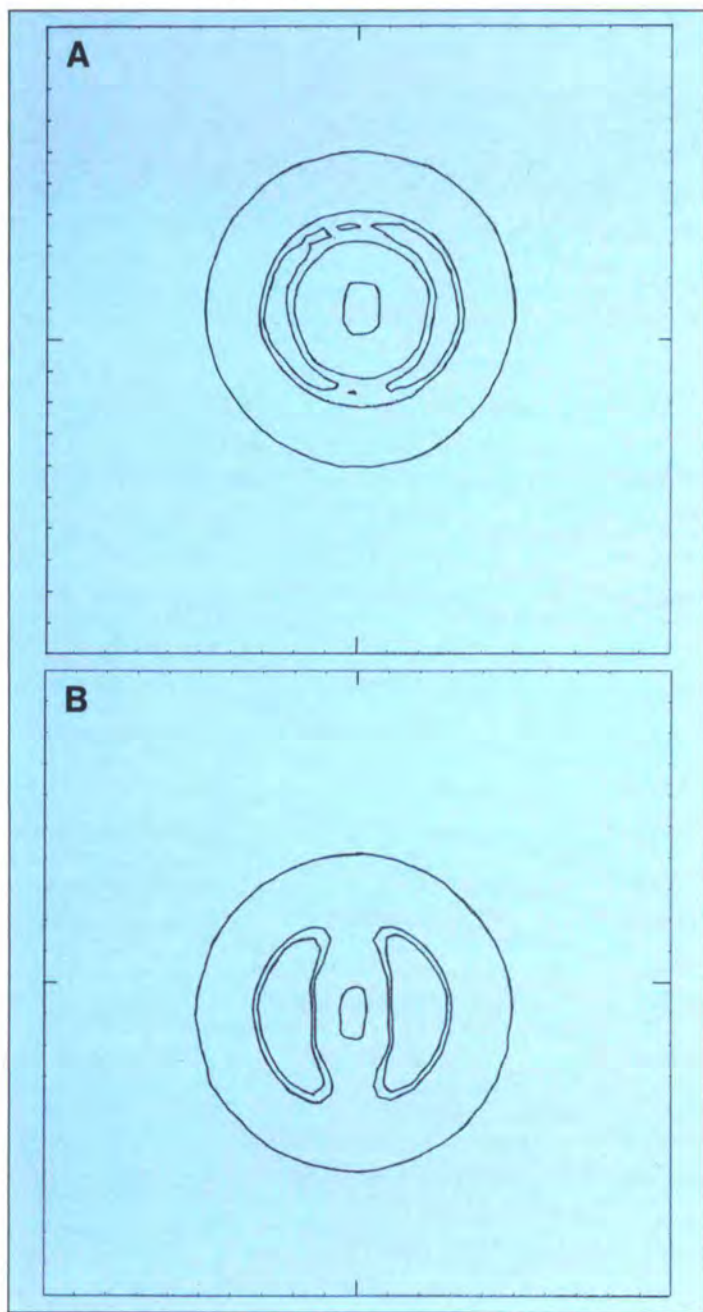


Figure 48: Contour plot of scattering; the pattern of 30% polymethyl methacrylate stabilized by poly-12-hydroxy stearic acid in dodecane (a) static, (b) sheared at  $10.000 \text{ s}^{-1}$ .

## Dynamics of Polymers

A recent trend has been towards the use of time resolved elastic scattering as a supplement to inelastic and quasi-elastic scattering for studies of dynamics of polymer molecules. The large

size of these molecules and the rather long relaxation times have restricted the value of conventional measurements as regards the study of concentrated polymeric systems.

A novel experiment exploiting the high flux has been conducted on a polycarbonate sample (Darmstadt, Mainz, ILL). Deuterated polymers with weak links were prepared - on heating these degrade in a controlled way yielding shorter polymers that then adjust to the equilibrium configuration for the lower molecular weight. A blend of 20% deuterated polymer allowed spectra to be recorded every 5 minutes on D17. This permits the changes in molecular weight, radius of gyration and conformation to be monitored. Some examples of data measured at  $190^\circ\text{C}$  are shown in Fig. 49 clearly indicating the variation of the time constant of the diffusion process with scattering vector. The experiment clearly shows the slow motion over long distances, the rapid segmental orientation and the constancy of the structure factor at the smallest distances.

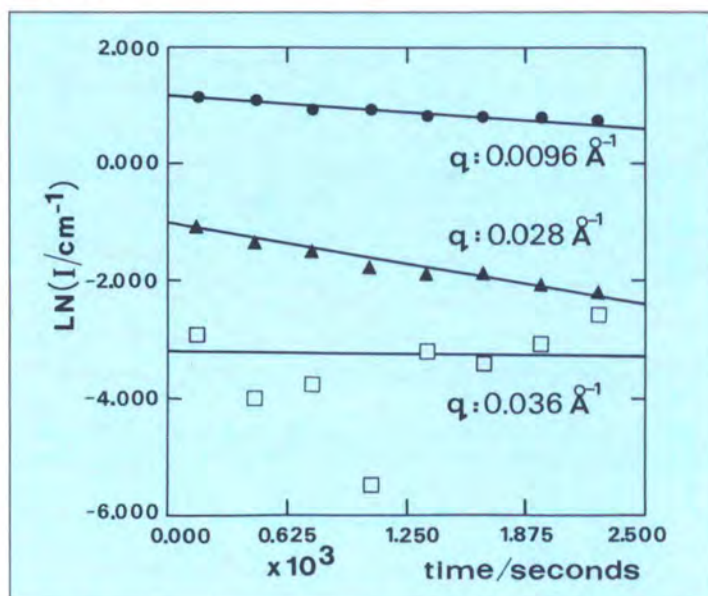


Figure 49: Logarithm of scattered intensity against time for three selected scattering vectors  $\odot q = 0.0096$ ,  $\triangle q = 0.028$ ,  $\square q = 0.036 \text{ \AA}^{-1}$ . The slope indicates the time constant of relaxation at the corresponding length scale.

Other work on dynamics has continued with the spin-echo spectrometer - a study of polyionomers (Imperial College, Exon) has explored a new class of materials. The work on structure and dynamics of elastomers (Strasbourg, Mainz, ILL) has continued on the small angle scattering spectrometer. For these experiments the in-beam stretching apparatus from the Universität Mainz has been employed. This unique equipment allows several centimetres strain to be applied at frequencies from 0.005 up to 10 Hz while the sample is in the neutron beam.

## Polymer Blends

Polymer blends have become a popular field of study as these materials gain commercial importance. Neutron techniques are exploited to determine the single chain structure factor and thus information about interaction parameters as well as observations of phase separation. Groups from Strathclyde and Mainz/Ludwigshafen have continued experiments in this field.

Secretaries: G. Kearley (9a)  
A. Rennie (9b)

## Polymer Relaxation in Solution and Melt Studied by Quasielastic Neutron Scattering

The main part of the quasielastic neutron scattering work in the field of polymers is centered around the investigation of large scale intramolecular relaxations. While the motion of single bonds along the backbone of a polymer chain is restrained by the details of the local potential, the longer range movements tend to become independent of such constraints and are thought to exhibit universal behaviour. Entropic forces resulting from the conformational entropy in the polymer free energy, hydrodynamic interactions between different polymer segments in solution and topological constraints or entanglements in concentrated systems govern the motional properties. The discovery of the equivalence between polymer conformations in solution and spin correlations in ferromagnets near  $T_c$  led to a transfer of ideas on many body effects in the theory of phase transitions and critical phenomena to the description of polymer systems. Finally the scaling ansatz was also applied to polymer dynamics [1].

Experimentally, this field has been opened up to neutron scattering by the application of the neutron spin-echo (NSE) technique. Uniquely, it provides the necessary energy resolution (20 neV) at sufficiently small scattering vectors ( $q \geq 0.02 \text{ \AA}^{-1}$ ). It is worth mentioning that NSE as a Fourier method measures directly the intermediate scattering function  $S(q,t)$ , a quantity of particular usefulness in the study of relaxation phenomena [2]. Experimental efforts commenced with studies on the motion on linear polymers in dilute solution, where internal relaxation is governed by the hydrodynamic interaction, i.e. the mutual interaction between polymer segments via the solvent backflow [3]. Under these circumstances the characteristic relaxation frequency  $\Omega(q)$  should scale with the third power of the scattering vector  $q$  (Zimm modes). Within the  $q$ - $t$  range of the spin-echo spectrometer IN11 at the ILL, such behaviour has been verified for several flexible polymer systems (PDMS, PMMA) in  $\theta$  as well as in good solvents [3,4]. Figure 50 presents a typical set of spectra taken on PDMS in Bromobenzene which is a  $\theta$ -solvent at 84 C. The solid lines are the results of a fit with the Zimm model which, using only one adjustable parameter, describes the data very well. Systematic deviations towards lower exponents for less flexible chains (PS, PTHF) are interpreted as resulting from the influence of the local potential. This provides a means of studying the backbone dynamics on a more local scale and of extracting information on the potential and local hindrance, a subject upon which current interest is focussed [5]. Important problems arise if the interaction between the segments is changed; the most conspicuous cases showing this phenomenon are the polyelectrolytes consisting of charged polymers (polyions) and counter-ions. The long range Coulomb interaction reduces coiling and leads to chain segments of large persistence length  $\ell_s$ . In the coupled poly-counter ion system slow chain relaxation and counter ion motion, which at small  $q$  eventually becomes a plasmon mode, are of importance. The chain relaxation has been found to be characterized by a cross-over from a strongly  $q$ -dependent diffusion coefficient for  $q\ell_s < 1$  to  $D(q) = \text{const}$  for  $q\ell_s > 1$  [6]. The plasmon mode is still to be verified. Due to the weak intensities involved, further experimental activities will greatly benefit from the envisag-

ed multidetector spin-echo system IN11B. Currently, more weakly interacting molecules such as polymers with dipolar interaction are being studied.

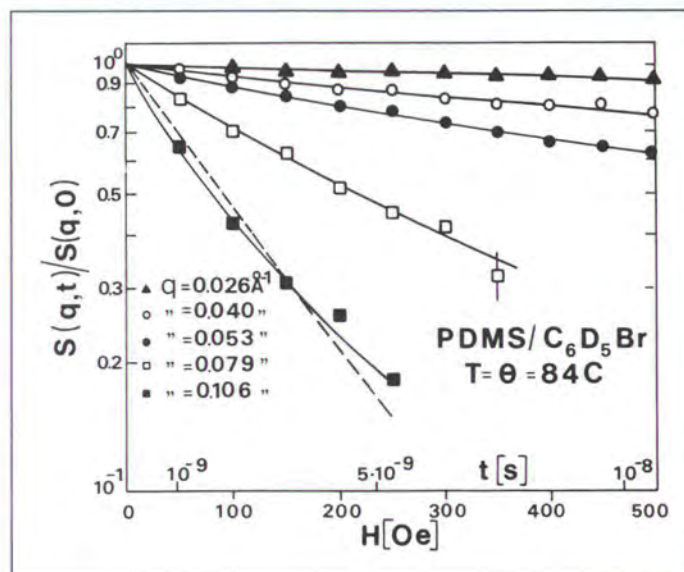


Figure 50: Relaxation spectra  $S(q,t)/S(q,0)$  as a function of the time for a dilute solution of PDMS in chlorobenzene taken at the  $\theta$ -temperature. Thereby  $S(q,t)/S(q,0)$  is given by the neutron polarization and  $t$  by the value of the guide field  $H$  in a spin echo set-up.

With increasing monomer concentration, different chains start overlapping and forming a transient network of mesh size  $\xi_c$ . With respect to chain dynamics both the collective response of the many chain ensemble as well as the single chain motion are of interest.

(I) *Many chain system:* From analogy with permanent swollen networks for spatial scales  $r > \xi_c$  a collective relaxation spectrum  $\Omega \sim D_c q^2$  is anticipated, while for shorter distances the network is not yet effective and  $\Omega \sim q^3$  should still be valid. Spatial cross-over between both régimes is expected for  $q\xi_c \approx 1$ . Quasielastic neutron scattering on PDMS in d-benzene led to a direct observation of this cross-over. Figure 51 summarizes the experimental results. While at low polymer concentrations the observed characteristic frequencies follow the  $q^3$ -law, with increasing concentration a cross-over to a  $q^2$ -behaviour is found at increasing  $q$ -values. The plateau values at low  $q$  thereby relate to the collective diffusion coefficient  $D_c$ , the cross-over points to  $\xi_c$ .  $\xi_c$  and  $D_c$  were found to scale with the monomer concentration according to the predictions of dynamical scaling [7].

(II) *Single Chain system:* For the relaxation of a single chain the screening of the hydrodynamic interaction is of prime importance. For distances longer than a hydrodynamic length  $\xi_H$  the backflow field is screened out and chain relaxation is thought to be controlled by the entropic forces alone. Spin-echo experiments on labelled H-PDMS chains in contrast matched semidilute PDMS solutions allowed a microscopic observation of hydrodynamic screening, i.e. a gradual cross-over from hydrodynamic modes (Zimm) to decoupled entropy controlled modes (Rouse), and led to a determination of  $\xi_H(c)$  [8]. The experimental result of nearly coinciding  $\xi_H$  and  $\xi_c$  is in favour of one of the basic assumptions of dynamical scaling. However, other than theoretically anticipated, the hydrodynamic screening is not complete, but

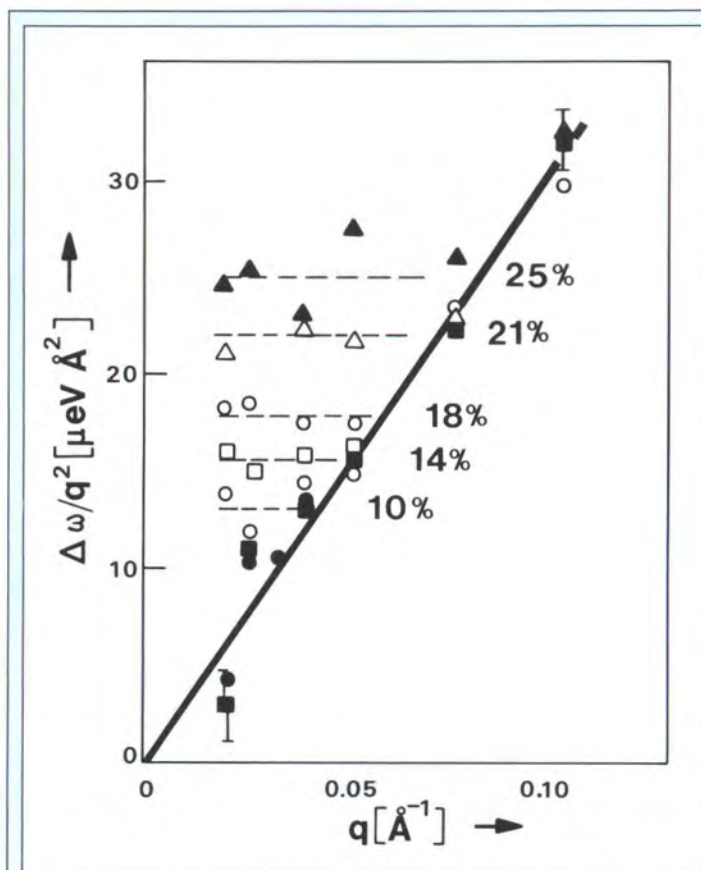


Figure 51: Concentration cross-over in PDMS/D-benzene. Characteristic frequencies  $\Delta\omega/q^2$  for different polymer concentrations at 70°C as a function of momentum transfer  $q$ . The solid line represents the low concentration  $q^3$ -dependence due to Zimm relaxation, the dashed lines show the  $q^2$ -behaviour characteristic for collective diffusion.

a residual time dependent viscosity remains which mediates the backflow field beyond  $\xi_H$ . Its microscopic explanation is still open.

In dense polymer systems the influence of entanglements is supposed to determine their dynamic properties. The experimental results on macroscopic diffusion and zero shear viscosity support the reptation idea. It postulates that due to mutual entanglements significant chain motions only occur inside a tube along the chain profile. These constraints should also influence the internal chain relaxation modes. Experiments undertaken on labelled PDMS [8,9] and PTHF [10] melts revealed similar results which, however, were interpreted in opposite ways. While the experimental line-shape seems to follow the local reptation model, its dependence on the scattering vector  $q$  disagrees strongly. So far a definite experimental answer on the existence of reptation on a microscopic scale cannot be given. Further experiments at smaller scattering vectors with better resolution, as will be possible on the new ultra high resolution spin-echo machine IN15, are required to solve the puzzle.

Besides the above-described work on linear polymers, more complicated systems such as star molecules, permanently linked gels and rubber networks are coming into focus. Most experimental work is still in an early stage and much of it suffers from the still limited  $q$ - $t$  range of IN11. With some exceptions, these complicated though very important macromolecules will only be reached by the projected IN15.

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**INSTRUMENT OPERATION DEPARTMENT**

## EINLEITUNG

Bekanntlich war das Jahr 1985 gekennzeichnet durch einen sehr langen Reaktorstillstand. Diese für die Experimente am ILL zwangsläufige Unterbrechung hatte eine Vielzahl von Arbeiten zur Folge, die von den verschiedenen Gruppen der Abteilung Instrumentenbetrieb ausgeführt werden mussten : Ab- und Wiederaufbauarbeiten sowie Neuaufbauten in den Experimentierhallen, Wiederinstandsetzung bestimmter Flüssigkeitsverteilungsnetze, Reparaturen und Verbesserungen der wissenschaftlichen Ausrüstungen (Instrumente und Probenumgebung), Entwicklung von Messeinrichtungen für sehr tiefe Temperaturen, Tests der Sicherheitskreisläufe, usw ...

Alle diese Arbeiten, die genau geplant und meistens vom Fortschritt der Arbeiten am Reaktor abhängig waren, konnten bis zum Wiederanfahren des Reaktors beendet werden. Dieser Zeitraum hat einmal mehr das Vorhandensein einer wirksamen Zusammenarbeit zwischen den verschiedenen Gruppen und den verschiedenen Mitarbeitern dieser Gruppen gezeigt.

In mehreren Sektoren sind Änderungen von Labors und Büros vorgenommen worden, um die Arbeitsbedingungen zu verbessern, und es werden jetzt Studien angestellt, um die mit der zunehmenden Benutzung von Rechnerendgeräten in den Büros verbundene Arbeitsumgebung zu verbessern.

## INTRODUCTION

The long reactor shutdown was the essential feature of the year 1985. This enforced interruption for the instruments at ILL, resulted in a variety of work to be dealt with by the various groups in the Instrument Operation Department : instrument dismantling followed by reconstruction, new instrument installations in the experimental halls, modernisation of certain fluid distribution circuits, repairs and improvements to scientific equipment (instruments and sample environment facilities), development of very low temperature measuring equipment, testing of safety circuits, etc.

All these operations, precisely scheduled and very often dependent on the progress of work on the reactor, were completed for the restart of the reactor. This period demonstrated once again the efficient collaboration existing between the various groups and staff within these groups.

Modifications of laboratories and offices have been undertaken in several sectors to improve working conditions, and studies are now being made to improve the working environment associated with the increased use of computer terminals in the offices.

## INTRODUCTION

L'année 1985 a été, on le sait, essentiellement celle d'un très long arrêt du réacteur. Cette pause, obligatoire pour les expériences à l'I.L.L., a eu pour conséquence une multitude de travaux auxquels les différents services du Département Exploitation des Instruments ont dû faire face : démontages puis remontages ou installations nouvelles dans les halls d'expériences, remise en état de certains circuits de distribution des fluides, réparations et améliorations des équipements scientifiques (instruments et environnement des échantillons), développement d'appareillages pour les mesures à très basses températures, tests des circuits de sécurité, etc...

Toutes ces opérations, rigoureusement planifiées et le plus souvent tributaires de l'état d'avancement des travaux sur le réacteur, ont pu être menées à bien pour la date du redémarrage. Cette période a montré une fois de plus l'existence de collaborations efficaces entre les différents services et les différents personnels de ces services.

Des modifications de laboratoires et bureaux ont été effectuées dans différents secteurs. Des études sont en cours afin d'améliorer les conditions de travail liées à l'utilisation croissante de terminaux dans les bureaux.

Au niveau des structures, certains regroupements simplificateurs ont

Bezüglich der Organisation der Abteilung Instrumentenbetrieb sind einige vereinfachende Neugliederungen vorgenommen worden. Die kleinen technischen Gruppen, die die Unterstützung für Vakuum, Tieftemperatureinrichtungen, Öfen, Hochdruck und verschiedene Dienstleistungen (wie z.B. Sicherheit, Transport, Reinigung, usw) an den Experimenten sicherstellen, sind jetzt in die Zentralgruppe für Unterstützung an den Experimenten eingegliedert. Die Arbeitsaufträge der Wissenschaftler sind so vereinfacht worden und können wirksamer ausgeführt werden, die Verantwortlichkeiten sind weniger verstreut als vorher, und die verfügbare Arbeitskraft wird den täglichen oder langfristigeren Dringlichkeiten folgend besser eingesetzt.

Die natürlichen Entwicklungen der Wissenschaft bewirken, dass die Anforderungen für die Probenumgebung an den Experimenten immer dringender werden und immer komplizierteren Einrichtungen entsprechen : tiefere (Bereiche der mK) und höhere Temperaturen, manchmal mit sehr schwierigen physikalisch-chemischen Bedingungen verbunden (z.B. Experimente an hochschmelzenden Übergangsmetallen in der flüssigen Phase), höherer Druck, starke Magnetfelder, zeitaufgelöste Messungen, usw. Es handelt sich hier ganz offensichtlich um einen kritischen Bereich, den man ohne Verzug berücksichtigen sollte, sowohl hinsichtlich des Budgets als auch der hochqualifizierten Arbeitskräfte, wenn die am ILL betriebene Wissenschaft auf ihrem höchsten Niveau bleiben soll.

As regards structures, certain re-organisations with a view to simplification have been carried out within the Instrument Operation Department. The small technical groups responsible for assistance on experiments for the problems of vacuum, cryogenics, furnaces, high pressures and miscellaneous intervention work (such as safety, handling, cleaning, etc) are now incorporated in a 'Service Central d'Assistance aux Expériences'. As a result the requests for intervention work from the scientists are simplified and more efficiently satisfied, the responsibilities are less dispersed and the available personnel will be better deployed to respond to urgent and longer term requirements.

The natural development of science results in the sample environment requirements on the experiments becoming increasingly demanding and requiring more sophisticated equipment : low temperatures in the mK range, high temperatures above 2000° C (in some cases associated with very difficult physical-chemical conditions, for example experiments on refractory transition metals in their liquid state), ever higher pressures, more intense magnetic fields, time-resolved measurements, etc. It is evident that this is a critical sector for which provision should be made as soon as possible, both as regards budget and highly qualified staff, if the science done at the ILL is to remain at its best possible level.

été effectués au sein du Département Exploitation des Instruments. Les petits groupes techniques chargés de l'assistance aux expériences pour les problèmes de vide, cryogénie, fours, hautes pressions et interventions diverses (telles que sécurité, manutention, nettoyage, etc...) sont maintenant intégrés dans un Service Central d'Assistance aux Expériences. Les demandes d'interventions de la part des scientifiques s'en trouvent simplifiées et plus efficacement satisfaites, les responsabilités sont moins diluées que par le passé et la main d'oeuvre disponible est mieux utilisée en fonction des urgences quotidiennes ou à plus long terme.

Les développements naturels de la science font que les demandes d'environnement des échantillons sur les expériences se font de plus en plus pressantes et correspondent à des équipements de plus en plus sophistiqués : températures de plus en plus basses (domaines des mK) et de plus en plus hautes, parfois associées à des conditions physico-chimiques très difficiles (expériences sur les métaux de transition réfractaires dans leur état liquide par exemple), pressions de plus en plus élevées, champs magnétiques intenses, mesures résolues dans le temps, etc... De toute évidence, il s'agit là d'un secteur critique qu'il faudra rapidement prendre en compte, à la fois sur le plan budgétaire et en main d'oeuvre hautement qualifiée, si l'on veut que la science faite à l'I.L.L. reste à son meilleur niveau.

# SPECIAL CRYOGENICS

## THERMOMETRY AND INSTRUMENTATION

The main aim this year was to introduce on a broad basis temperature controllers developed during previous years. The performances measured on a pre-production batch of 6 were as follows:

1 K	< T <	10 K	$\Delta T =$	$10^{-3}$ K
10 K	< T <	300 K	$\Delta T =$	$10^{-2}$ K
300 K	< T <	2 800 K	$\Delta T =$	0.1 K

Their great versatility (both semiconductor and metallic resistance thermometers and all types of thermocouples) has been checked experimentally.

The RS 232 links with the experiment computer operated rapidly and reliably.

In fact, 20 controllers were ordered at the end of 1984, 10 from the firm which produced the pre-production batch of 6, and 10 from a local subcontractor.

In the event the original firm was unable to complete the order satisfactorily, this contract was cancelled and finally placed with the local sub-contractor, which caused a delivery delay of 6 months.

The 6 controllers in the pre-production batch plus the first ten controllers under the subsequent order, have been successfully installed on 15 instruments. They operate as specified, with a completely automatic temperature control using the instrument computer. It is now possible for an experimentalist to verify and change the temperature from home.

The performance and convenience of these controllers have led to requests to supply them to other institutes in France, Germany and Denmark.

The transfer of know-how associated with the change of manufacturer resulted in a heavy load on the thermometry laboratory, which was nevertheless able to deal with the following additional work :

- Provision on a general basis of calibrated thermometers (platinum, rhodium-iron, carbon, vitreous carbon, germanium) which can be supplied to ILL users within a few minutes.
- Maintenance of helium level gauges.
- Stock control.
- Miscellaneous assistance.

## SUPERCONDUCTING MAGNETS

The 6T cryomagnet (Oxford Instruments) for the Triple Axis Group was finally delivered in July, with reduced performance (transition at 4.35 T). Negotiations are in progress for reconstruction of the coils. In the meantime the system is being used in its present state.

The variable temperature insert of the 7.4 T cryomagnet (Thor) had to be rebuilt following an implosion.

Considerable assistance was given to the scientists on D3 with the modification and improvement of the variable temperature insert for the 4.8 Tesla cryomagnet.

The ILL now has 5 cryomagnets :

Instrument	H/Tesla	Gap/mm	Diam/mm
D3	4.8	15	16
D2	5 (horiz.)	50	19
3 axis	4	38	50
General use	7.3	15	35
General use	10	40	35

## VERY LOW TEMPERATURES

The CRTBT-ILL dilution refrigerator has achieved the anticipated performance (10 mK in 5 hours). It has been used continuously since the reactor re-start, including during the reactor shutdowns, for preparatory measurements.

The construction of 5 'Neumaier' type dilution inserts has been undertaken (2 for the National Bureau of Standards, 1 for the Rutherford Laboratory, and 2 for ILL). This construction work, which should have been completed up to the end of 1985, has been delayed by two months.

Two pumping units for dilution refrigerators have been built. The demand for very low temperature measurements is increasing very rapidly :

1984 : 70 days for 4 cycles.

1985 : 115 days for 1.5 cycles.

This result could not have been obtained without the assistance and close collaboration of the scientists concerned.

We have also twice had to ask for the assistance of the CRTBT for the loan of an additional cryostat (with the staff to operate it).

The number of actual measuring days at very low temperatures between 3 September and 25 November 1985 was as follows:

- 52 days (8 samples), with the CRTBT-ILL dilution refrigerator.
- 32 days (5 samples), with the 50 mm diameter insert.
- 21 days (1 sample) with the He3 cryostat.
- 10 days (2 samples) with the dilution refrigerator loaned by the CRTBT.

We have already had 3 very low temperature cryostats in simultaneous operation and we shall have up to 4 in the near future. This will result in a serious overload for the group concerned because of the particular difficulty of this type of experiment.

## MISCELLANEOUS

During the shutdown, a cold valve driven by a vapour pressure bulb (freon 12) was developed. It is currently undergoing longterm tests on two instruments before being made widely available. Current results are very satisfactory.

## CENTRAL SERVICE

### GENERAL INTERVENTIONS GROUP

The role of the group remains that of aiding the smooth operation of the experimental program, and has involved close liaison with the scientific staff and visitors and the supervision of the experimental zones from the point of view of safe operation - particularly the surveillance of hazardous samples, (PuTe, PbSb, Np, etc), the regular testing of the instrument safety interlocks, the use of the SADI alarm system, and the responsibility for all transport and handling within the experimental halls.

### TEMPERATURE AND PRESSURE GROUP

Repairs and modifications have been made to the standard cryostats on request, and maintenance has been carried out on the pool of liquid helium and nitrogen reservoirs, including the equipping of the majority of the helium dewars with digital readout gauges.

The expenditure on liquid helium has continued to be a preoccupation this year, despite the reduced consumption.

The recovery system has been thoroughly checked and the leak rate reduced. An argon gas supply has been installed, and will soon be fully operational, permitting the use of argon gas instead of the more costly helium gas for flight paths.

In order to make users aware of the quantities of liquid helium used and gas lost, monthly returns are now established and distributed for each experimental position.

#### *Consumption : 1.12.84 - 31.08.85 (reactor shut-down)*

During this period liquid helium was used only in the laboratories and for the testing of special cryogenic installations, except for the months of July and August when the reservoirs were re-cooled in preparation for the start-up.

Liquid helium consumed	6 826 litres
Gas returned to CENG	1 758 m3
Liquid Nitrogen consumed	131 050 litres

#### *Consumption 1.09.1985 - 30.11.1985 (reactor operating)*

Liquid helium consumed	17 447 litres
Gas recovered	10 866 m3

of this recovered gas 1.945 m3 (15 %) was supplied to the instruments for the flushing and filling of flight paths and 8.471 m3 returned to the CENG.

Liquid Nitrogen consumed	96 273 litres
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### High Temperature

Since the start-up of the reactor, furnaces have been regularly in use on the instruments in the temperature range 200-1600° C. Following development by the Group the "3-Axis furnace" and control unit equipped with a programmable CERBERE control unit is in operation, as also is a miniature furnace for use with quartz cells on D17 in the range 50 to 250° C. The group has assembled a 1600° C furnace for IN13, and participated in the assembly and operation of a furnace for the growing of single crystals.

Improvements have been made to the standard 3-Axis furnace following measurements of its temperature gradient and, top loading furnaces operating up to 1600° C are under construction. A new furnace has been designed for D11 and D17.

Under development is a furnace with improved screening, which has already been tested to 2600° C.

### High Pressures

New aluminium alloy pressure vessels have been designed and tested for use up to  $P = 5$  Kbar and calibrations of manganin pressure gauges have been checked in order to ensure a better reproducibility of experiments. Pressure cells of clamp-type (cf picture) have been tested and used in collaboration with scientists at CEN-G. Further collaboration has been established first with CEN-G to build pressure cells which fit into a cryomagnet and second, with Laboratoire Léon Brillouin at Saclay to implement a Helium gas pressure system for neutron scattering experiments.

Further developments include the design of more compact 30 Kbar-pressure cells and the realization of a uniaxial stress device which permits experiments at low temperature under high magnetic field.

### VACUUM GROUP

Interventions and maintenance during the year included the revision of more than 100 primary and secondary pumps, oil changing on 250 pumps, bearing changes on 35 pump and motor units, and 400 helium leak tests.

25 mobile helium pumping sets for general use were constructed or refurbished and new pumping sets constructed for the guides. Vacuum tests were carried out on the reactor thimbles before and after installation, and on the guides after realignment.



Pressure cell of the clamp type.

# MAINTENANCE AND EQUIPMENT OF BUILDINGS AND EXPERIMENTAL POSITIONS

## MAINTENANCE WORK

The main purpose is to keep in an operational state all the technical installations and buildings, apart from the reactor. The principal work in 1985 has included the reconstruction of the air conditioning installation for the computer room, the installation of experimental cabins and their conditioning systems, the replacement of electrical power supplies for the experimental instruments, and the weather proofing of the roof of ILL8.

## CONSTRUCTION AND EQUIPMENT WORK

The group continued in its role of collaboration with the General Intervention Group, the Reactor Department and the Instruments and Methods Department, to define and organise

the construction and assembly of the biological shielding and other installations (electricity, air conditioning, handling, infrastructure) required in the experimental halls, and in its role of defining and designing building modifications and technical constructions.

## TECHNICAL ASSISTANCE TO OTHER GROUPS

### Workshop services

The self service mechanical workshop was available for ILL staff to permit them to modify or make parts at short notice. The main sheet metal workshop produced items which were difficult to subcontract (prototypes, items requiring fitting on the spot, or components required urgently).

### Services for external organisations

This concerns the European Molecular Laboratory (EMBL) which has an outstation on the ILL site. The group carried out, under contract, maintenance of their buildings and technical installations, their power supplies and any work required in the modification or equipment of their laboratories.

### General Services

The Group was involved in provision of logistical support (display panels, etc) for special visits and events in the ILL life, it also carried out the majority of office and laboratory removals.

# INSTRUMENT GROUP FUNDAMENTAL AND NUCLEAR PHYSICS

- PN1: Fission product separator (LOHENGRIN) on beam tube H8 (H.R. Faust, J.P. Bocquet, G. Martinez, I. Gartshore).
- PN2: Beta spectrometer (BILL) on the vertical beam tube V3 (K. Schreckenbach, G. Colvin, G. Blanc).
- 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, F. Hoyler, S. Robinson).
- PN4: Ge(Li) pair spectrometer on beam tube H7 (F. Hoyler).
- SN5: Ultra-cold (UCN) and very cold (VCN) neutron source with 3 beam positions on the inclined beam tube IH3 (P. Ageron, W. Mampe, A. Beynet, T. Manning).
- SN6: On-line mass separator for thermally ionized fission products (OSTIS) on neutron guide H23L (U. Stöhlker, T. Manning).
- SN7: Cold polarized neutron beam at end position of guide H14 (K. Schreckenbach, W. Mampe).
- PN8: Fission product coincidence spectrometer (COSI FAN TUTTE), (P. Geltenbort, F. Gönnewein, T. Manning).
- H17: Cold neutron guide with liquid helium UCN source (P. Ageron, Ch. Jewel).

- H18: Cold neutron guide (W. Mampe).
- H22: Thermal neutron guide: neutron induced particle emission (H22D), J.P. Bocquet); neutron induced fission (H22E), (J.P. Bocquet);  $\gamma$ - $\gamma$  angular correlations (H22F), (F. Hoyler).
- TGV: Vertical VCN guide in cold source connected to Steyerl turbine (Niveau D), (P. Ageron, W. Drexel, W. Mampe, A. Beynet, A. Steyerl).

## INTRODUCTION

For College 3 activity during the year 1985 was strongly determined by the remaining 8 months of the reactor stop until the end of August. The following activities were related to this event: (a) 3 beam tubes out of 5 (H6-H7, IH1, IH3) involving extended environmental construction work were exchanged by the reactor team; (b) major repairs, improvements and innovations on the existing instruments have been performed; (c) 3 new facilities (TGV, Gams 4,  $\lambda > 20 \text{ \AA}$  diffractometer) have been installed; (d) the background situation and the experimental areas for some instruments were improved; (e) an intense programme of ILL staff visits to other laboratories - for the first time in 'reverse direction' - was profitable for the preparation of future experiments at ILL and technology transfer. As from September the performance of the rebuilt and improved instruments was tested and the normal external user experiments were resumed.

On the personnel side the following changes have taken place: S. Robinson joined PN3, K. Schreckenbach took over respon-

sibilities in the SN7 area, F. Gönnerwein finished a 6-year period as College 3 Senior Scientist. The vacancy has now been filled by D. Dubbers, coming from Heidelberg.

## INSTRUMENT IMPROVEMENTS AND MODIFICATIONS

### Fission Research

**LOHENGRIN (PN1):** The high voltage was increased to 380 KV on both plates of the condenser by cleaning internal parts (which improved the vacuum by a factor of 6) and by changing most of the high voltage insulators. Furthermore a cryo pump installed in the experimental section increased the quality of the vacuum sufficiently to facilitate the use of large thin windows at the spectrometer exit (accompanied by important gas diffusion) without having problems with the formation process of the condenser plates. These thin windows together with a new flange system at the separator exit, which allows for adjustments to the focal plane within 72 cm of parabola length, make it possible to use thick covered  $^{235}\text{U}$ ,  $^{239}\text{U}$  and  $^{241}\text{Am}$  sources for nuclear spectroscopy work without the risk of beam tube contamination. This ensures that high sensitivity measurements such as yields in cold fragmentation, yields in symmetry and nuclear charge will not suffer from background perturbations caused by sputtered material from thick sources.

The large ionisation chamber was prepared for installation with a divided anode for  $E \cdot \Delta E$  measurements (to fix the nuclear charge of the fission products) and with a delay line read out of the x position along the parabola. During the last two years efforts were undertaken to obtain and to prepare actinides for fission studies on LOHENGRIN. Up to now the following targets can be proposed to the user community:  $^{233}\text{U}$ ,  $^{235}\text{U}$ ,  $^{239}\text{Pu}$ ,  $^{241}\text{Pu}$ ,  $^{237}\text{Np}(2n, f)$ ,  $^{241}\text{Am}(2n, f)$ ,  $^{244}\text{Cm}(2n, f)$ ,  $^{229}\text{Th}$ ,  $^{249}\text{Cf}$ .

**COSI FAN TUTTE (PN8):** Due to the exchange of the beam-tube IH1, the fission fragment coincidence spectrometer had to be removed and was provisionally installed in the guide hall. A series of improvements was performed in the meantime: (a) the gas supply system has been modified to reduce contamination of the ionisation chamber counting gas by  $\text{O}_2$  and  $\text{N}_2$ . (b) After installing a differential pumping system between the beam tube and the high vacuum chamber of the spectrometer thin Al-foils ( $20 \mu\text{gr. cm}^{-2}$ ) can be used to separate these sections, leading to an important reduction in  $\gamma$ -background. (c) A reliable control system has been designed and put into operation.

(d) A new software package was installed for the treatment of up to eight-parameter measurements. This permits on-line evaluation of energy and velocity distributions of the fission fragments as well as mass distribution.

(e) Determination of the nuclear charge of light fission products is now a standard feature of the spectrometer. This was achieved by optimizing the ratio track length/Frisch grid-anode distance as well as by the use of well adapted linear amplifiers.

**OSTIS (SN6):** Most of the components of OSTIS were repaired and improved during the shut-down. This work was completed by the adjustment of the separator and the remeasurement of its most important characteristics: the transmission was found to be about 90% with a mass resolution greater than 750. The data acquisition system was improved by adding a recently developed multiscaling controller unit and off-line tests have been performed to optimize the negative ion source.

### Spectroscopy

Three improvements to the smooth running of the electron spectrometer BILL (PN2) have been accomplished: (i) the outdated PDP11/10 has been replaced by a more powerful PDP11/23. (ii) A more precise neutron monitoring system consisting of three independent neutron detectors around the beam tube have been installed. This configuration, by averaging the 3 count rates, eliminates the count rate errors observed with a single counter when the beam-tube is slightly flexed. (iii) An improved handling of "hot" targets and target disposal has been accomplished by an extension to the present glove box system.

Finally and for the future, the construction of a 64-wire proportional counter is now being envisaged.

The crystal spectrometers GAMS1 and GAMS4 (PN3) and the pair spectrometer PN4, which all had to be completely disassembled to enable the exchange of the throughgoing beam tube H6-H7 (Spring 1985), have been reconstructed and are again fully operational. During this reconstruction period the opportunity was taken to make several important improvements to the instruments.

(A) In close collaboration with the reactor department a new source holder tube has been installed. Figure 52 shows a schematic drawing of the new arrangement and the photograph shows the front section of the source holder tube into which the targets are loaded. Whereas the former source holder had only one target position (corresponding to No. 2 in Fig. 52) the new holder offers 5 different positions. Position 2 (which is adjustable) is used for targets which are to be viewed by the bent-crystal spectrometers GAMS 1,2,3. Positions 1 and 3 will be used in addition to position 2 when additional quantities of target material are needed for the double flat crystal spectrometer GAMS4 or for the pair-spectrometer PN4. Positions 4 and 5 are 'breeding positions' which can be used to produce certain unstable targets in order to investigate more exotic nuclei by triple neutron capture.

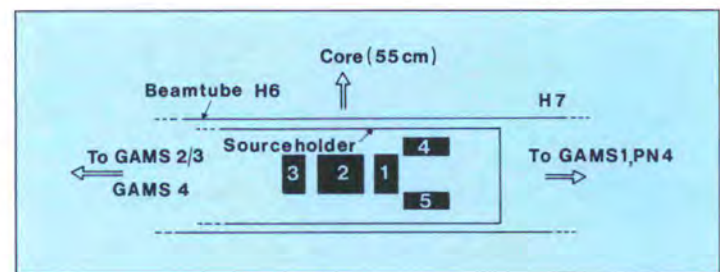


Figure 52: Schematic drawing of the different target positions

In addition the source holder tube can be placed in a second irradiation position, where the target is 75 cm from the reactor core. In this position the neutron flux at the target is 2.4 times lower than in the primary position ( $d = 55 \text{ cm}$ ) and thus comparative measurements can be made for the same target in two different neutron fluxes. This comparison enables the direct assignment of isotopes produced after single and double neutron capture since the relevant gamma transition intensities scale with the flux for single capture but with the square of the flux for double capture (Fig. 53) (when the cross-sections are compatible). This change of target position means that optimum resolution for the best crystal spectrometers will be obtained for crystals with a different radius of curvature

than those currently in use. A holder for bent crystals enabling a variable selection of radii has been constructed and is undergoing tests.

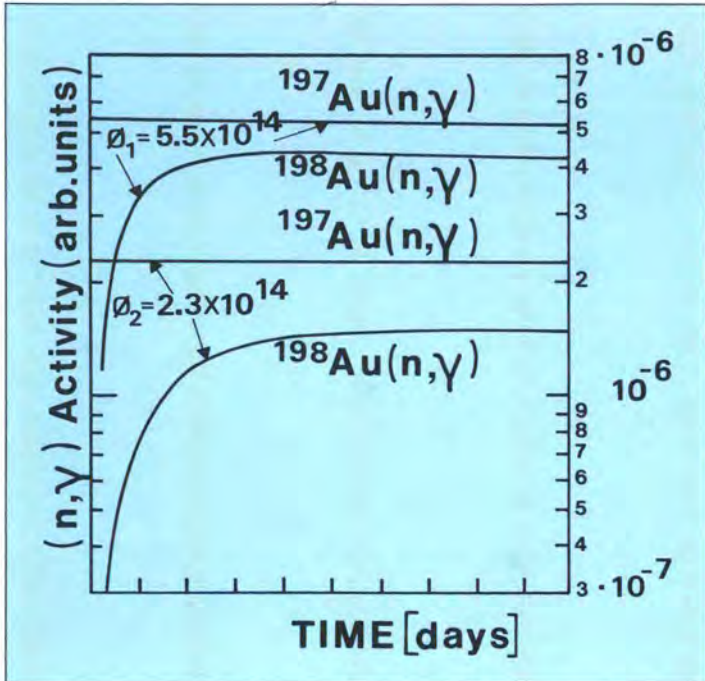


Figure 53: Single and Double neutron capture on a  $^{197}\text{Au}$  target for two different neutron fluxes (calculated)

A big improvement has been obtained in the mechanical stability of sources under irradiation. Figure 54 compares source movements measured with GAMS1 before the modification (Plot A,  $^{190}\text{Os}(n,\gamma)$  - Sept. 1984) with a recent measurement (Plot B,  $^{197}\text{Au}(n,\gamma)$  - Nov. 1985). The effect of this improvement is that the optimum resolution of  $\sim 0.6''$  (G2) previously obtained only under ideal conditions for very short time

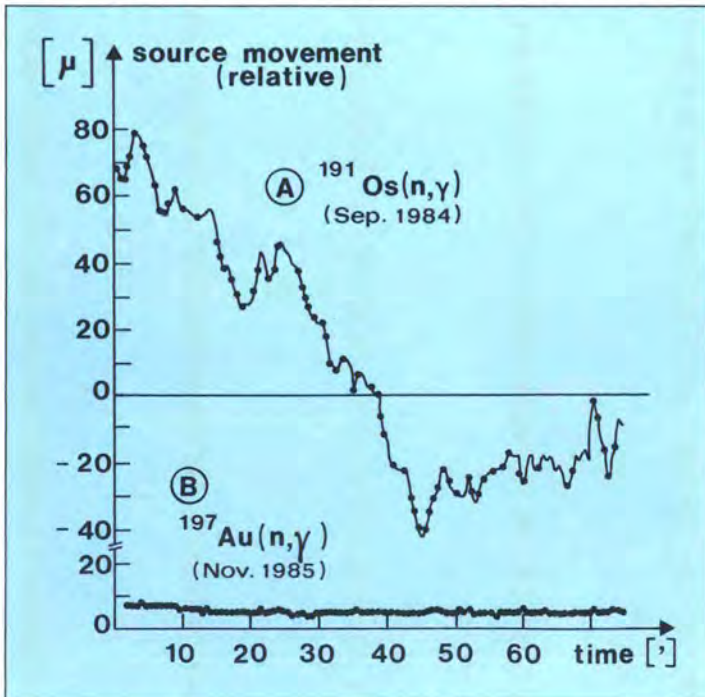


Figure 54: Relative movement between target and crystal, measured with the control system of GAMS1.  
A: before reactor shut-down 1984;  
B: after reactor shut-down 1985.

spans, is now standard and can be maintained for the whole duration of an experimental run. Figure 55 shows a portion of a recently measured spectrum from  $^{197}\text{Au}(n,\gamma)$  on GAMS1.

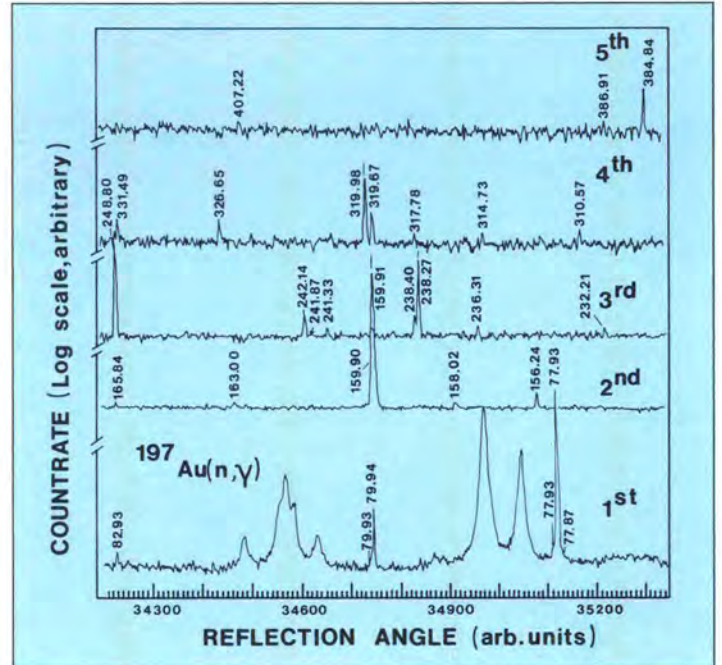


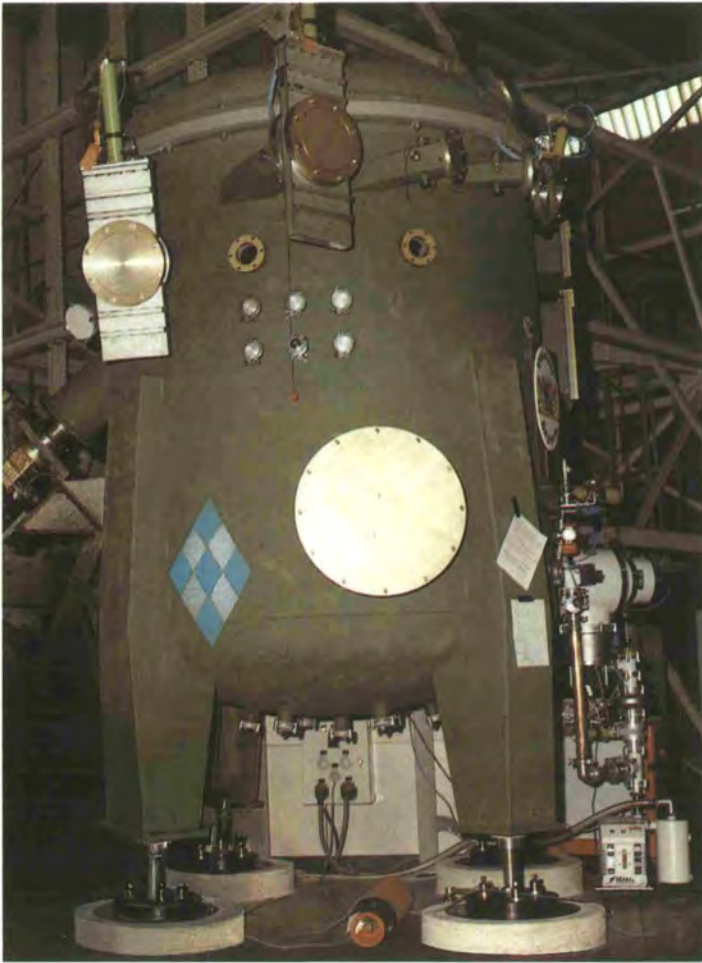
Figure 55: Portion of a  $^{197}\text{Au}(n,\gamma)$  spectrum, measured in the first five reflection orders simultaneously. The large peaks correspond to X-rays whose natural line width is bigger than the resolving power of the spectrometer. Notice: The scale is logarithmic!

(B) The GAMS1 data acquisition system has been modified by a combined effort of the GAMS instrument group and the I.L.L. software development group. The data are now taken via an ADC and in an on-line software fit windows are set such that the count rates for the different orders of reflection can be determined. This allows for the simultaneous recording of up to 10 orders of reflection whereas previously only 5 orders were available. It is hoped that the purchase of a new computer for GAMS 2,3 in the near future will provide the conditions for the installation of a more flexible system on this instrument showing similar improvements.

### Fundamental Physics

ULTRA COLD NEUTRONS - SN5: (a) All the instruments together with the platform had to be removed for the exchange of the beam tube IH3. On the same occasion the degraded in-pile source section of the UCN source had been replaced. (b) The experiment searching for an electric dipole moment (EDM) of the neutron has been reinstalled on SN5 since the superthermal He source was not operational on the beam H17 in 1985. The previous computer was replaced by a VAX (RAL) and the performance of the three integrated Rb magnetometers significantly improved by the Sussex group. (c) A stainless steel UCN storage system has been set up for density measurements. (d) A computer controlled  $\theta$ - $2\theta$  diffractometer has been designed, constructed and installed on the VCN beam of SN5 for general reflectivity measurements (see Section 'Special Studies of Experimental and Instrumental Techniques' for more detail).

NEW VERTICAL VCN-UCN SOURCE(T-TGV): Together with the modified vertical cold source, the 5 m long in-pile guide and vacuum tube of the TGV and the related neutron shutter have been installed by the reactor department. The curved,



The UCN-turbine at the niveau D of the reactor (status December 1985). The neutron guide extracting very cold neutrons from the new vertical Cold Source can be seen on the left. The platform for the various experimental set-ups is not yet mounted in the picture.

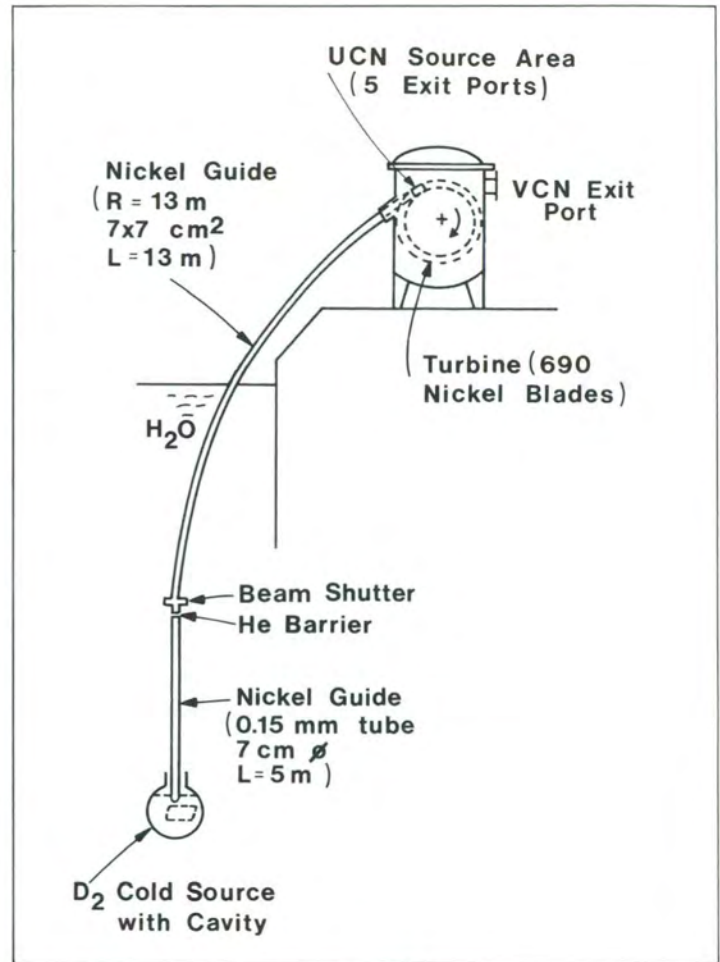
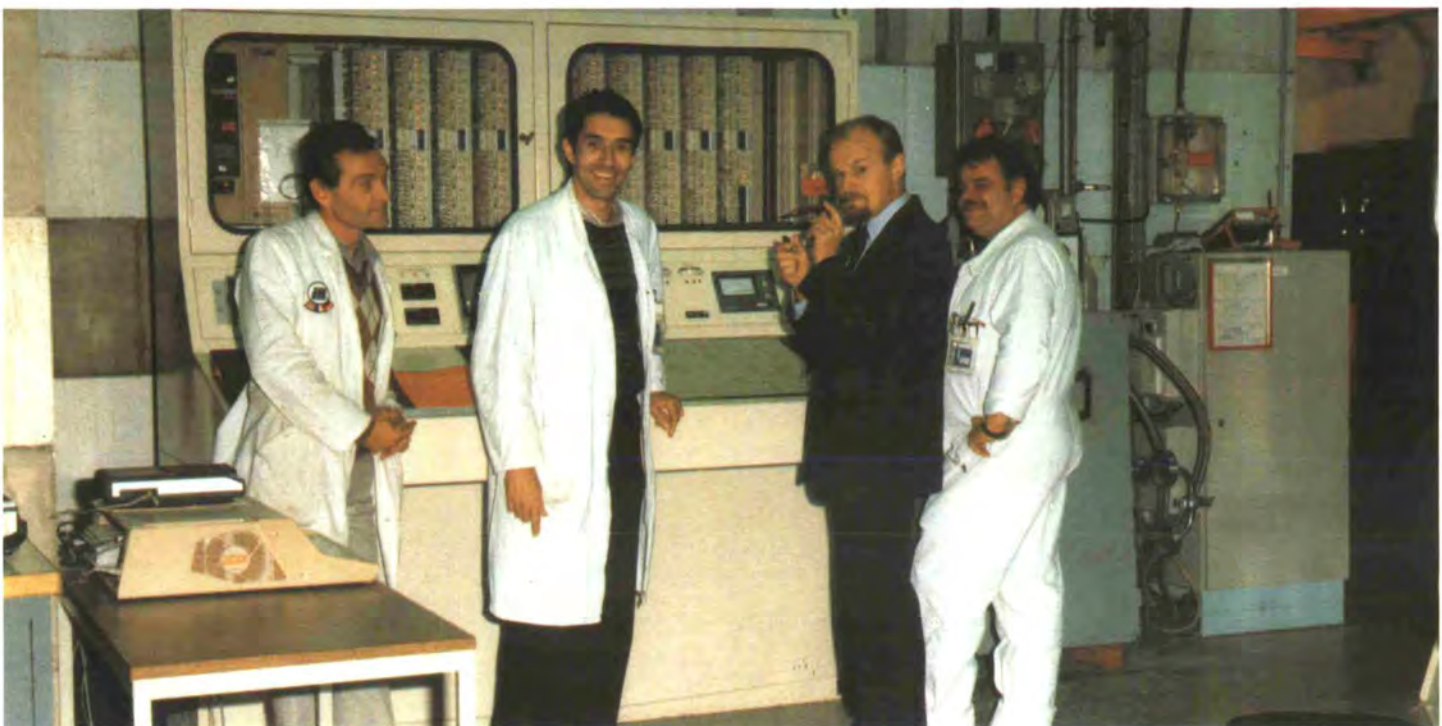


Figure 56: Schematic view of the new VCN-UCN source.



The picture shows the control panel for the PN3 source changing device, together with the reactor staff who was extremely efficient and competent in designing and installing the new source holder tube.



Front part of the new target holder of PN3.

all metal guide section of  $7 \times 7 \text{ cm}^2$  cross-section, built at Garching, was installed in the swimming pool towards the end of the reactor stop. A standard bottle for UCN density measurements and a time of flight set-up have been used to measure the performance of this new high reflectivity guide. The guide is connected to the Garching turbine. The vacuum system for the guide and the turbine are in place and a  $50 \text{ m}^2$  platform 164 cm high is being installed in order to adapt the experiments to the beams, 330 cm above the floor level. One  $3 \times 7 \text{ cm}^2$  VCN beam and 4 UCN beams will be available in 1986 together with the first generation of instruments. (For more details see section 'Special Studies of Experimental and Instrumental Techniques'). (see Fig. 56)

#### COLD NEUTRON BEAMS

The area of the H18 beam has been prepared for the refined version of the Bayreuth neutron charge experiment. The instrument is currently being set up. The polarized beam position SN7 has been equipped with an air conditioned experimental area outside the first neutron guide hall. The beam has been temporarily equipped with a velocity selector for experiments with PERKEO which was improved and reinstalled in 1985.

Concluding, we remark that all the instruments of College 3 have been considerably improved during the reactor stop and resumed their operation on schedule.

Co-ordinator: W. Mampe

## INSTRUMENT GROUP THREE-AXIS SPECTROMETERS

- IN1 : 3-axis and beryllium-filter spectrometer on the hot beam-tube H8 (U. Steigenberger, B. Dorner, H.J. Lauter, J.P. Varini).
- IN3 : 3-axis spectrometer on the thermal guide-tube H24 (H.J. Lauter, R. Arthaud).
- IN8 : 3-axis spectrometer on the thermal beam-tube H10 (R. Currat, P. Frings, C. Vettier, A. Brochier).
- IN12: 3-axis spectrometer on the cold guide-tube H142 (W.G. Stirling, K.A. McEwen, D. Puschner).
- IN14: 3-axis spectrometer on a cold guide-tube on the second cold source (W.G. Stirling, W. Kaiser).
- IN20: 3-axis spectrometer with polarized neutron option on the thermal beam-tube H13 (R. Pynn, E. Lindley, W. Kaiser, P. Flores).

### IN1: Hot source 3-axis spectrometer

In 1985, the major improvement to the hot-source triple-axis spectrometer IN1 was the installation of a new mechanical support for the monochromator system; this now allows better optimisation of the alignment of the monochromators. The sample goniometer was equipped with motors and coders, controlled by the instrument computer. Motors were also added to diaphragms and to the support of the silicon (fast-neutron) filter. To alleviate the problems of coexistence between the three instruments using the H8 beam-tube, the marble 'Tanzboden' floor was extended; the interchange of secondary spectrometers is now much simpler and faster, and testing of 'off-duty' instruments can be carried out.

After the successful installation of a PDP 11/73 computer on IN8, it is intended to continue the three-axis modernisation program by replacing the IN1 Solar computer with a PDP 11/73 early in 1986.

### IN1: Be-filter

The Be-filter spectrometer continued to operate satisfactorily, despite some problems with the  $2\theta_M$  movement which now seem to be resolved. As well as the conventional Be-filter, a combination Be-pyrolytic graphite filter is available, providing  $\Delta E/E_0$  of about 2% up to  $E_0 = 45 \text{ THz}$ . This is particularly useful for strongly scattering samples. It is intended to study new designs for both filters, with improved fast-neutron shielding and increased detector solid angle to give a higher signal.

### IN3: 3-axis spectrometer on thermal guide

Despite recurring problems with the control electronics and the Tanzboden step-motors, all scheduled experiments on the IN3 spectrometer were carried out. In 1986, the control computer will be replaced by a PDP 11/73 and the electronics modified to permit closed-loop control of D.C. motors for the Tanzboden movements. A new double-faced monochromator (P.G.(002) and Cu(111)) will be installed. Both monochromators will have variable vertical curvature.

A change of the position of IN3 is envisaged. By use of the end position of the H25 guide, the instrument would gain in flux and flexibility, while using the existing Tanzboden floor. The instrument IN13, which is situated after IN3 on the H24 guide, would also be improved as part of this project.

## IN8: 3-axis spectrometer on thermal beam H10

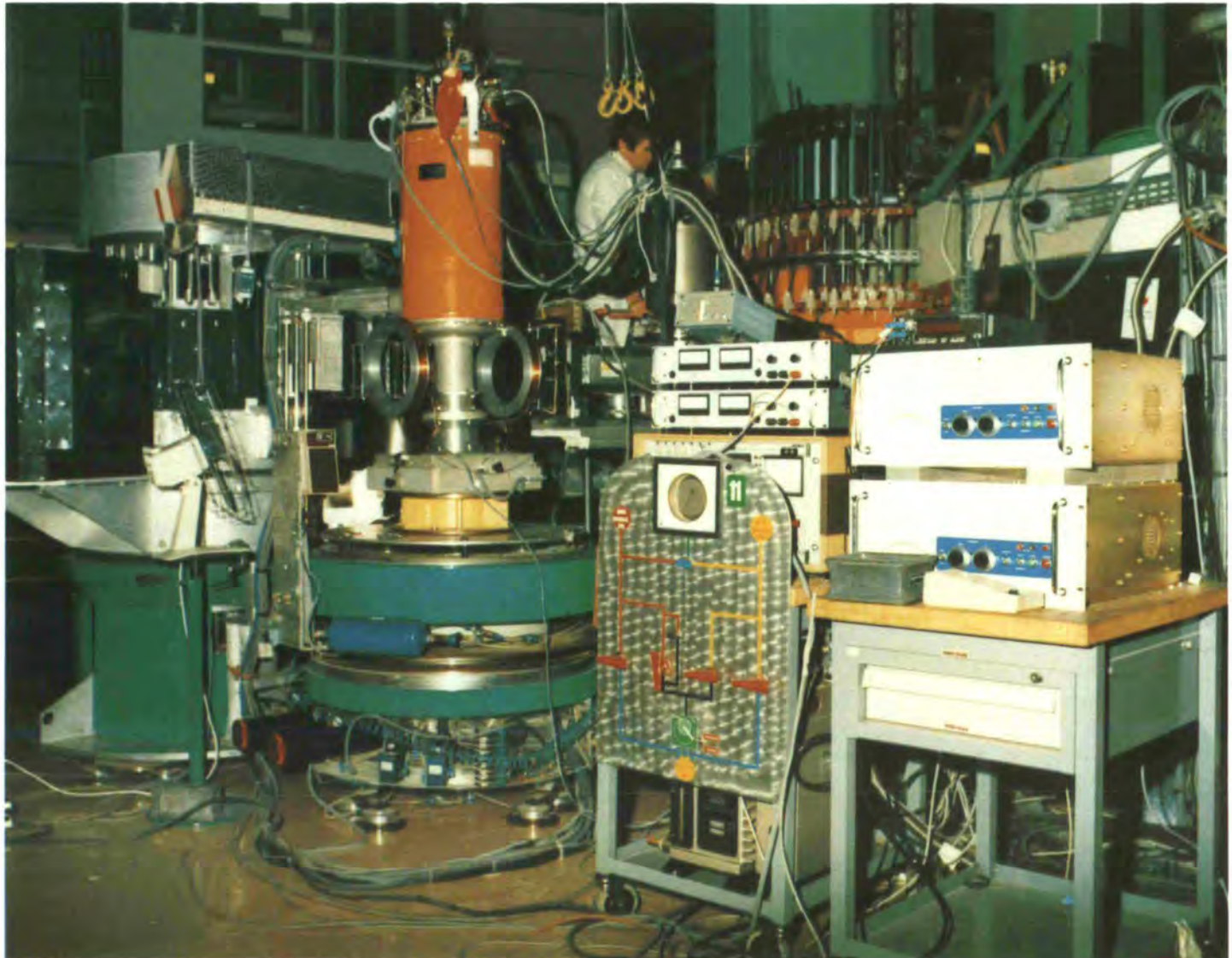
During the long shutdown the primary and monochromatic beam geometry of IN8 was modified in order to take advantage of the increased height of the new vertically-focussing monochromator crystals. As a result of the larger vertical acceptance, a twofold flux gain was achieved at the sample position. For example, at  $\lambda = 1.34 \text{ \AA}$ , with  $40'$  collimation, the monochromatic flux is now  $2.2 \cdot 10^8 \text{ n/cm}^2/\text{s}$  using pyrolytic graphite (002) and  $1.3 \cdot 10^8 \text{ n/cm}^2/\text{s}$  using Cu(111) as monochromator crystals. A vertically-focussing Cu(220) monochromator is at present under construction and will be installed before the end of the year.

In parallel, a new PDP 11/73 instrument computer was installed as a replacement for the Solar 16/40. The new operating system is now almost identical to that of the DEC-10 and data

files are produced in ASCII format. The spectrometer control program is much more flexible, as almost every operation, including spectrometer and sample alignment, can be done automatically. The status of the machine can be checked from any MICOM-terminal. This new software, currently in use on IN20 and IN8, is expected to become the future 3-axis spectrometer standard.

## IN12: 3-axis spectrometer on cold-neutron guide

The cold-neutron spectrometer IN12 is now equipped with a fully automatic, large angle ( $-139 < 2\theta_{\text{A}} < +142$ ) analyser shielding system. This has simplified operation of the instrument and the background is significantly improved ( $< 0.5$  counts per minute with a beam of  $15 \text{ cm}^2$ ). IN12 continues to be used frequently in its polarized neutron mode; improvements to the polarized beam equipment continue. For the future, the replacement of the Solar computer is planned, as on the other instruments of the group. The existing electronics will eventually be replaced by the closed-loop/D.C. motor system being developed for IN14.



IN12 set-up for polarization analysis experiments. Neutrons are polarized by a super mirror after the graphite monochromator. Polarization analysis is carried out with a Heusler crystal analyser. The ancillary power supplies for the magnetic field coils and flippers can be seen in the foreground.

### **IN20: 3-axis spectrometer with polarization analysis on thermal beam H13**

During the reactor shutdown the IN20 (polarized beam, thermal triple-axis spectrometer) site was prepared; the new casemate was built and part of the existing marble floor was replaced. In parallel with this work, construction of spectrometer components continued. Shortly after the September start-up, the monochromator drum was installed and its efficiency as biological shielding was tested with entirely satisfactory results. Mechanical and electronic tests continue and the first test experiment is planned for early in 1986.

### **IN14: 3-axis spectrometer for 2 - 6 Å neutrons**

Although pressure of work on the reactor and existing instruments slowed down somewhat the development of the IN14 project, with the restart of the reactor, design work has accelerated. This instrument was accorded the highest priority by the Instrument Subcommittee meeting of September 1985. Invitations to tender for the monochromator shielding will be sent out early in 1986 with construction of the instrument in the Hall d'Essais planned for 1987. The motor drive system - D.C. motors operated in closed-loop configuration - and data-acquisition electronics are at present under development. The current estimate is that the instrument will be ready for first test operation late in 1987.

Coordinator: W. G. Stirling

## **INSTRUMENT GROUP TIME-OF-FLIGHT, HIGH RESOLUTION AND DIFFUSE SCATTERING**

- IN4 : Time-of-flight spectrometer on thermal tube H12 (A. Murani, W. Langel, A. Key).
- IN5 : Multi-chopper spectrometer on cold guide H16 (F. Douchin, G. Kearley, H. Blank, S. Jenkins).
- IN6 : Focusing TOF spectrometer on cold guide H15 (A.J. Dianoux, M. Bée, Y. Blanc).
- IN10 : Backscattering spectrometer on cold guide H15 (A. Magerl, C. Poinignon, H. Godfrin, P. Joubert).
- IN11 : Spin-echo spectrometer on cold guide H141 (B. Sarkisian, B. Farago, J.P. Varini).
- IN13 : Backscattering spectrometer for short wavelengths on thermal guide H24 (W. Petry, A. Heidemann, J.F. Barthelemy).
- D7 : Diffuse scattering spectrometer on cold guide H15 (O. Schärpf, W. Just, C. Ritter, R. Rebesco).
- D11 : Small angle and diffuse scattering spectrometer on cold guide H15 (P. Timmins, A. Wright, R. May, R. Baker).
- D17 : Low Q, High Resolution spectrometer (R. May, J. Torbet, P. Timmins, M. Cruz).

### **IN4: TOF spectrometer on thermal tube H12**

The new Fermi chopper rotating on magnetic bearings, installed in August 1984, is now working satisfactorily. At its maximum speed of 30,000 r.p.m., the width of the neutron pulse is  $\approx 7 \mu\text{s}$ . Modifications to the secondary spectrometer, to reduce the flight path, have now to be decided.

### **IN5: TOF spectrometer on cold guide H16**

The renewal of the IN5 primary spectrometer was completed early in November. It includes the magnetic bearings, suspension of the choppers and new driving and phasing systems, both developed at KFA-Jülich. The suspension is electrically controlled in the axial direction, while being inactive in the radial directions. This results in very small power consumption and maximum mechanical safety (see fig. 57). The associated computer programs, as well as some other parts of the system ('pick-ups', safety devices etc.) were developed at the ILL. The whole 'multi-chopper' can now be controlled automatically as a function of the experimental parameters ( $\lambda_0$  etc.) with the exception of the TOF electronics for which the control system is being completed. The rotational speed (20,000 r.p.m) and the phase stability obtained were in perfect agreement with the expected values. The flux decrease due to the chopper acceleration (1.6 - 0.79) is largely compensated

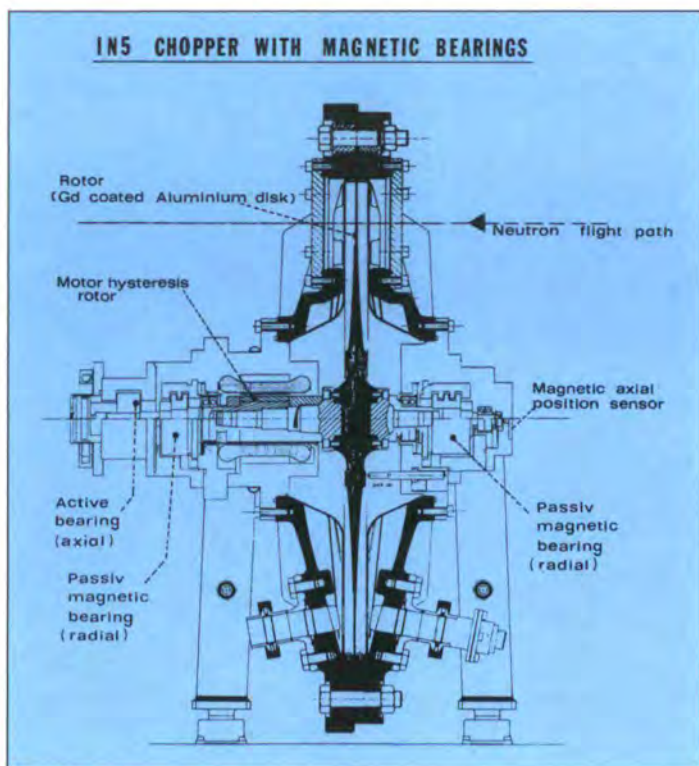


Figure 57: New chopper system for IN5.

for by the flux increase due to the improvement of the Cold Source and the realignment of the guides. Other improvements in progress are:

- Mounting of a first (small) area of fixed elliptic detectors at large angle
- Direct connection of fixed detectors at small angles
- Connection of the flight path box to the Argon supply
- Cryostat improvements (faster temperature variation)
- New graphic terminal (shared with IN6)

The following improvements are planned by summer 1986:

- Oscillating collimator (background reduction) mounted in a new sample tank (February)
- Full computer control of the TOF unit
- Computer control of the sample temperature by a standard ILL cryostat control unit
- New cryostat for very low temperature inserts.

## IN6: TOF spectrometer on cold guide H15

The stability of the detectors has been improved during the long shutdown by improved shielding against dust in the detector boxes; dust can cause flashes of the high voltage supply and thus lead to electronic noise. Another effort has been made to reduce the background (sample independent) of the instrument, which was already rather low; this is in view of exceedingly demanding experiments such as the one on the measurement of excitations in liquid He<sup>3</sup>. Another shielding by Cd around the choppers and the Be-filter has been installed and all the possible entrance paths of spurious neutrons have been blocked by boronated plastic. The results can be summarized by 4 numbers giving the total counting rate on the 337 detectors (i.e. 3.04 m<sup>2</sup> of active detection surface):

- Reactor stopped 0.28 c/s
- Reactor at 57 MW, beam off 0.51 c/s
- Reactor at 57 MW, beam on but stopped at the entrance of sample box ( $\lambda = 5.1 \text{ \AA}$ ) 4.7 c/s
- Reactor at 57 MW, beam on, impinging on a vanadium plate,

$\varnothing 5 \text{ cm}$ , thickness 1 mm ( $\lambda = 5.1 \text{ \AA}$ ) 1495 c/s. At  $\lambda = 5.1 \text{ \AA}$  the flux of the instrument has been increased by a factor of 1.75 in comparison with the flux before the long shutdown: this results from the excellent design of the new vertical cold source, and presumably from the cleaning of the guides.

Control of the diaphragm, which is placed after the monochromator, is now operational; one can use a reduced beam size for certain types of experiments requiring, for example, a better Q-resolution. A new fast pumping unit has been installed which allows operation of the furnace at high temperature shortly after changing the sample (~ 1 hour).

The ILL precision temperature controller can be used to regulate the temperature of the cryostat, the furnace and the heating and cooling loop; this is achieved by connecting various power-amplifiers to the controller. The setting and the control of the temperature can now be programmed on the instrument terminal.

## IN10: Backscattering spectrometer on cold guide

IN10 had a good start after the long shutdown. An increase of the neutron flux by a factor of 1.7 was noted due to the modification of the Cold Source. However, after the installation of the monochromator of DB21, this gain factor dropped to 1.4.

A new monochromator based on a Si<sub>x</sub>Ge<sub>1-x</sub> crystal, with an offset of 12  $\mu\text{eV}$  has become available, enabling measurements of high resolution spectra including the elastic line up to 27  $\mu\text{eV}$ .

## IN11: Spin-echo spectrometer at the cold guide H141

The present major limitation on performance of IN11 is the use of a single detector of rather small solid angle, which severely limits the data collection rate. A multiple detector bank (46 detectors) with a wide angle magnet (at different scattering angles  $2\theta = 12^\circ$ , in steps of  $2\theta = 0.5^\circ$ ) has been installed. Associated correction coils,  $\pi/2$ -flippers and a collimator have already been designed. The existing computer and electronics will remain unchanged. The mirrors for polarization analysis will be CoTi supermirrors (of the type used on D7). The instrument is intended to be used for studies of dynamics of polymers and critical dynamics in magnetic and non-magnetic systems of extremely small energy changes (order of magnitude 0.01  $\mu\text{eV}$ ).

Tests were carried out during which some new fundamental ideas and changes were developed. The results indicate that the multidetector NSE spectrometer can be constructed successfully. The improved instrument is expected to be available by the end of 1986. The data collection rate will be increased by at least 40 times.

## IN13: Backscattering spectrometer for short wavelengths on thermal guide H24

IN13 was fully operational on Friday, 30 August 1985, the day the first neutrons were delivered to the guide H24. The flux at the sample position, measured with the IN13 monitor, is about the same as before the long reactor shutdown.

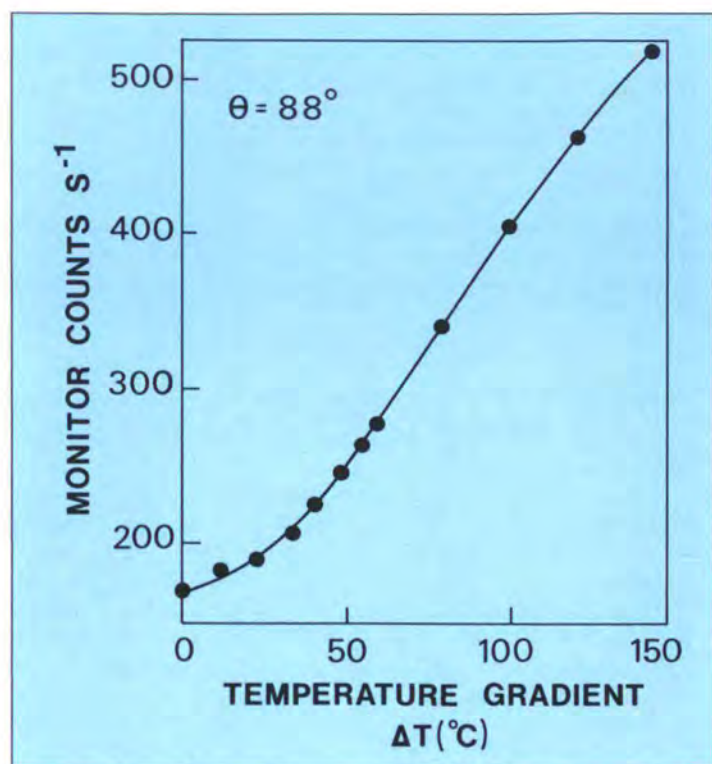


Figure 58: Monitor counts at the sample position of IN13 as a function of  $\Delta T$  for  $\theta_M = 88^\circ$

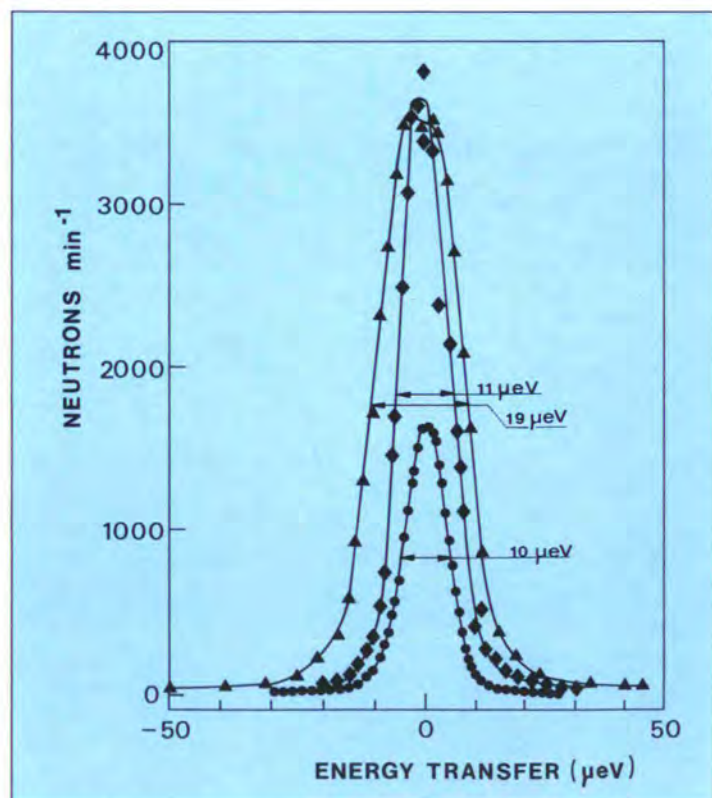


Figure 59: Energy scans with a standard vanadium scatterer on IN13 at  $\Delta T = 0$  (circles),  $59^\circ C$  (losanges) and  $128^\circ C$  (triangles). The intensities are normalised.

For the first time, a full reactor cycle has been successfully operated in a 'three-axis mode'. In this mode the energy variation of the incoming neutrons is performed by variation of the backscattering angle  $\theta_M$  at a fixed monochromator

temperature instead of changing the monochromator temperature at a fixed  $\theta_M$ . This permits a variation of the lattice spacing  $d$  by applying a temperature gradient on the monochromator crystals parallel to the neutron beam. The increased  $\Delta d/d$  gives rise to a higher flux at the sample position by slightly coarsening the energy resolution. Details are shown in Fig. 58 and Fig. 59. The software of IN13 has been improved to scan the monochromator and deflector angles in constant energy steps.

The construction of a combined monochromator cryostat and furnace has been completed and now permits a temperature variation of the  $CaF_2$  crystals between  $-196^\circ C$  and  $500^\circ C$ . First tests have shown excellent temperature stability and negligible temperature gradients over the whole monochromator volume of  $66\text{ cm}^3$ . This cryo-furnace will be operational in 1986 with energy scans from about  $-150\text{ }\mu eV$  to  $400\text{ }\mu eV$  at  $\theta_M = 89^\circ$ , i.e. an energy resolution of  $7-9\text{ }\mu eV$ . A project has been proposed to increase the flux of IN13 further by changing the height of the IN13 beam from  $12.5\text{ cm}$  to  $20\text{ cm}$ . The project foresees the move of the three-axis instrument IN3 from its position in front of IN13 to an end position on the neighbouring guide, thereby freeing the lower third of the guide H24 and essentially increasing the beam area available for IN13.

## D7: Diffuse scattering instrument

Major improvements have been implemented on D7.

### Gain of intensity

A flux gain of 1.7 has been noted due to the installation of the improved vertical Cold Source. As a consequence of the increase of intensity, the measuring times have become considerably shorter and thus the operating staff of D7 has necessarily to cope with an additional work load. Some users have already made use of the small detector bank (16 detectors of  $1.2\text{ cm}$   $\varnothing$ , fixed at the side of each movable bank) resulting in Q-resolutions somewhat between that of the former D7 and SAS instrument D11. In January this bank will be tested for polarization analysis with better Q-resolution by placing it behind two analysers.

### New chopper

To avoid any magnetic material in the beam, a chopper has been installed which uses air cushions as bearings. The disc material is a plastic mixed with Gd-oxide. In this way, depolarization effects, caused by eddy currents from the use of metal discs rotating in the magnetic guide field, can be avoided.

### Analyser improvements

About half of the 31 analysers are now equipped with mirrors having a super-mirror coating on both sides; this results in a considerable improvement of the transmitted polarized neutron beam (by a  $1.5\text{ cm}$   $\varnothing$  sample) of up to 50% compared with 20 - 30% previously obtained. This is important mainly for TOF measurements with polarization analysis.

A liquid Na experiment was conducted at  $840\text{ K}$  and  $\lambda = 3.13\text{ \AA}$  and  $4.79\text{ \AA}$  to check the performance of the set-up for spin polarization analysis at short wave-lengths. One analyzer was installed with supermirrors coated on both sides and a  $22.5\text{ m}$  radius of curvature. About 200 supermirrors are required

for such an analyser which yields a transmission of 15% for the elastic peak (sample of 1.5 cm  $\varnothing$  at 1.2 m distance from the polarizer entrance). Analysers with 100 supermirrors and a 10 m radius of curvature yield only 5% transmission of the elastic peak. One complete bank will be equipped with this improved analyser version.

### **D11: Small angle scattering diffractometer on cold guide H15**

D11 has been extensively rebuilt during 1985. The original sample-detector flight tube has been replaced by a new one of 1.6 m diameter. Instead of the five discrete detector positions previously available, the detector may now be placed at any distance from 1 to 37 metres from the sample. The detector is mounted on a trolley, whose movement is under computer control, allowing the full length of the tube to be covered in about 12 minutes.

The vacuum in the flight tube is maintained permanently and the only intervention necessary by the user is at the sample position where the vacuum is controlled by a single On/Off switch. The sample position is now much more open than before, facilitating the installation of cryostats and other special sample environments. In the coming months it is foreseen to make further improvements here with a system of beam-defining diaphragms to replace the iris, and also the purchase of a precise x,y,z, $\omega$  table. A standard ILL temperature controller has been ordered.

The detector electronics have been rebuilt and are installed on the back of the detector in a volume maintained at room temperature. Data transfer to the CAMAC is via a fibre optic link. The beam stop x,y movement is automated and computer controlled. There is also an option for automatically exchanging beam stops of different dimensions.

A VAX 11/730 has been installed as the instrument control computer, replacing the ten year old PDP 11/40. The increased memory allows for more flexible on-line data reduction and the increased disk space permits storage of several weeks raw data.

### **D17: Low Q, high resolution spectrometer**

New encoders have been installed in conjunction with ILL standard closed-loop modules for controlling the detector angle, beam-stop, sample-changer and Eulerion cradle  $\Omega$  movements. These replacements required a simultaneous adaptation of the data acquisition programs, which now allow a better choice of automatic measuring cycles. A more complete modernisation of the data acquisition software is planned at the time when the more than 10 years old PDP-11 computer is replaced by a VAX-730 in early 1986.

The pumping system has been completely overhauled; the pumps have been grouped together and now act separately on the three different volumes. The pump control system had to be modified accordingly.

The user's cabin has been separated from the electronics and computer by sliding doors. Control buttons for non-automated functions have been moved close to the console terminal.

Coordinators:  
M. Bée  
R. May



*The completed set-up of the new Small Angle Scattering facility D11. The diameter of the flight tube is 1.6 m and the detector may now be placed at any distance from 1 to 37 m from the sample.*

# INSTRUMENT GROUP DIFFRACTION INSTRUMENTS

- D1A : High resolution powder diffractometer on thermal guide H22 (A.Hewat).
- D1B : Two-axis diffractometer with multidetector on thermal guide H22 (A. Fitch, K. Ben Saidane).
- D2B : Very high resolution powder diffractometer on thermal beam H11 (A. Hewat, J. Davies).
- D3 : Two-axis polarized neutron diffractometer with lifting counter on thermal beam H5 (F. Tasset, J. Baruchel, A. Dorn).
- D4 : Liquids diffractometer sharing the hot beam H8 with IN1B (P. Chieux, R. Meyer, A. Hawes).
- D5 : Three-axis polarization analysis spectrometer on hot beam H4 (K. Ziebeck, J. Schweizer, P. Agnes).
- D9 : Four-circle diffractometer on hot beam H3 (W. Kuhs, M. Lehmann, J. Archer).
- D10 : Four-circle triple-axis spectrometer on thermal guide H24 (neutron spin echo option) (C. Zeyen, G. McIntyre, R. Chagnon).
- T12 : Neutron camera on thermal guide H23 (A. Wright).
- D15 : Four-circle MK6 diffractometer on the inclined thermal beam IH4 (J. Brown, S. Wilson, G. Schmid).
- D16 : Four-circle MK6 diffractometer on cold guide H16 (G. Zaccai, L. Braganza, J.-R. Reynal).
- D19B : Multidetector diffractometer for protein crystallography (S.A. Mason, M. Thomas, M. Berneron).
- D20 : High flux multidetector (in preparation) (J. Pannetier, P. Convert, J. Torregrossa).
- DB21 : Four-circle diffractometer with PSD for biological macromolecules (M. Roth, A. Bentley, G. Bentley, M. Thomas).
- S18 : Neutron interferometer on thermal neutron guide H25 (T. Wroblewski, A. Rumpf, G. Schmid).
- S20 : Neutron topography diffractometer (J. Baruchel).
- S21 : Double crystal diffractometer (C. Zeyen, R. Chagnon).
- S42 : Laue Diffractometer (J. Marmeggi, W. Drexel).
- S42B : Laue Diffractometer (J. Baruchel, A. Filhol).

## MAJOR INSTRUMENT MODIFICATIONS

### D1A, High Resolution Powder Diffractometer on H22 guide

D1A is replaced by D2B as the scheduled instrument, but will continue as a special beam machine with support from outside ILL. First such use in 1986 will include studies of stress in metals (6 weeks plus 2 weeks) and the structure of ice at high pressure (2 weeks). The demand for such use is already much greater than the time available. ILL will repair instrument faults, but technical, and hopefully financial, support must be provided by the user.

The machine itself has been largely rebuilt to reduce maintenance, with new electronics, new computer system (recovered from D8), new motors and new monochromator protection. Together with the new ILL temperature controller, these changes have also simplified the operation, making it easier for example to scan through temperature.

Unfortunately, the new monochromator protection was not installed during the long shutdown, and the temporary modifications needed to interchange the positions of the D1A and D1B monochromators resulted in a flux loss of more than 50% on D1A. This situation cannot be rectified before April 1986.

### D2B, Very High Resolution Powder Diffractometer on H11

D2B, sharing the H11 reactor beam tube with D19 and D20, replaces D1A as the scheduled instrument. The beam tube protection, which was mounted during the long shutdown, is very successful: at the monochromator position the beam is uniform with the expected intensity, and the background in the experimental area is low. The monochromator, 300 mm high vertically focussing squashed germanium, is the largest yet made at ILL, consisting of 28 individual 10 x 10 x 50 mm slices of crystal. The mechanics permit precise positioning of the 160° detector bank to within  $\pm 0.005^\circ$ . The PDP 11/24 computer system and electronics are to the latest ILL standard design, and as identical as possible to the new D1A system.

D2B will be capable of scanning through temperature more quickly than on D1A, since a full 160° pattern can be obtained with only 100 detector steps. Sample environment has therefore been given high priority. The new Dagleish ILL temperature controller, together with a new cryofurnace covering the range 1.5K to 250°C will be particularly important.

The machine was essentially complete in November 1985, but commissioning tests depend to some extent on progress on the other machines on the H11 beam. In any case, D2B must be 100% operational from February 1986 when D1A will be taken over for the Harwell special beam experiment.

### D1B, Two-axis diffractometer with multidetector on thermal guide H22

During the long shutdown a number of improvements have been made to D1B:

- The instrument and monochromator have been moved 40 cm towards the reactor, to allow the interchange in the monochromator positions in D1A and D1B without a change in the take-off angle. It is hoped that this will result in greater stability in the incident flux.

- A focussing graphite monochromator has been installed leading to an increase in incident flux by a factor of 4-5 at 2.511Å. Kinetic measurements can now be performed with a time resolution of the order of a few seconds.

- An ILL (programmable) precision temperature controller has been bought, accurate to 0.01°.

- The layout of the experimental area has been improved. The modifications to the casemate and the monochromator have led to some shielding problems, so that the shorter wavelength of 1.28Å is temporarily unavailable. These problems should be solved early in 1986.

## D3, Two-axis polarized neutron diffractometer on thermal beam H5

The previous software system made it difficult to implement further developments necessary for the efficient control of this diffractometer. A new system (RSX11M) has therefore been introduced and has become very popular within the Diffraction Group.

Changes of the 'users interface' of D3 have been kept to a minimum. The remote control has been facilitated and better service is provided now for data transmission and software maintenance and development.

Modifications were carried out on the 4.6 Tesla cryomagnet insert with variable temperature. A good stability has been attained even at the lowest accessible temperature (1.4K). The modification includes a separate He gas injection circuit hooked onto an internal heat exchanger which provides clean liquid He, disregarding the quality of the main liquid He bath. In this way the amount required for the cooling of the sample is precisely regulated by means of a programmable closed loop system including a Pirani pressure gauge and a newly developed automatic cold valve. The fine temperature reading and control is ensured by an ILL high precision regulator linked to the instrument computer.

## D4, Liquids Diffractometer on the Hot Beam H8

The D4 data acquisition system has been fully renewed. Although the MACAMAC control of the instrument had been a real success, it was not adapted to the complexity of the machine. The instrument is now directly controlled by the PDP 11/23 with two drive units to cope with the high data acquisition rate. The new MAD program for powder diffractometers has been installed and adapted to the particularities of the two multidetector scans. It allows for elaborate data acquisition strategies. A fully automatic control of temperature scans at low temperature has been successfully implemented and was very much appreciated by the exhausted experimentalists.

However, the machine itself requires a lot of further development. One multidetector, maintained by a considerable effort of special software developments for its control, was finally sent for repairs. Monitoring is not yet satisfactory. Sample environment design is still too elaborate and not adapted to the fast turnover rate of the instrument. Data analysis is far from being at a quality level comparable to the present statistical accuracy. In addition, we are still lacking the feedback from experienced users. The several years of interruption of the availability of the spectrometer has had a dramatic effect on the various research programmes and their neutron aspects from which we still need to recover.

## D5, Three axis polarisation analysis spectrometer on hot beam H4

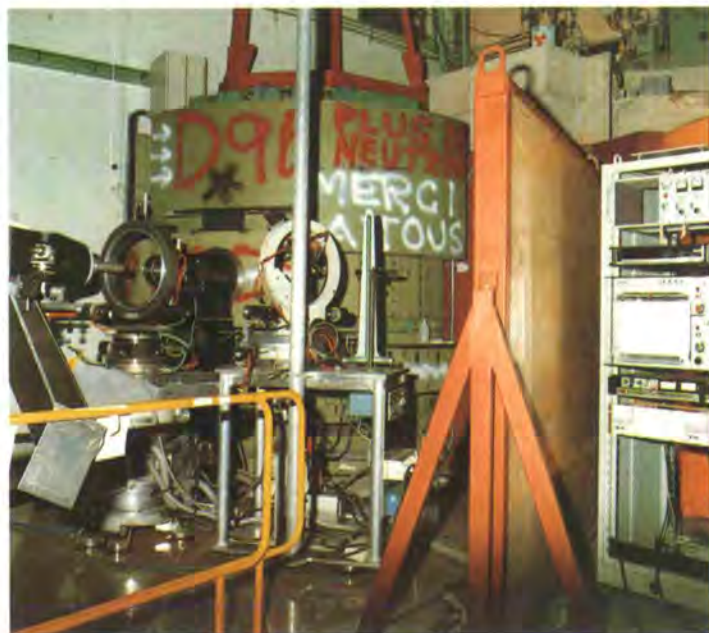
During the long shutdown D5 was completely dismantled to enable the in-pile beam tube to be changed. This action enabled the mechanics of D5 to be completely overhauled and certain bearings to be replaced. All motors and encoders were checked and the entire spectrometer was recabled. In addition, the monochromator shielding was substantially modified to facilitate the eventual replacement of D5 by D3. The modification to the monochromator shielding has reduced the torque

and enabled a new monochromator assembly to be installed. Each monochromator is now contained in a separate cassette which can be physically interchanged in a reproducible manner. The associated electronics and motors provide a more reliable and precise possibility of alignment. To accommodate the new motors and encoders it was necessary to modify both the electronics and the software. In spite of the major nature of the modifications to D5 the work was completed before the start-up of the reactor without any loss of beam time.

## D9B, Four-circle diffractometer on hot beam H3

The completely reconstructed four-circle diffractometer on the hot source should be operational well before the time of publishing this report. It is worthwhile to recall some of the major improvements with respect to the old D9. First measurements have shown that moving the instrument closer to the source gave the expected flux gain; the neutron flux at the sample position will be  $>10^7 \text{ ncm}^{-2} \text{ sec}^{-1}$ . The Cu monochromator working in transmission will be replaced by a horizontally focussing Be(110) monochromator which will help to increase the resolution at high scattering angles. The instrument, running with new motor control units, will be equipped with a 2-stage duplex cryorefrigerator which reaches temperatures down to 15K.

Some time in 1986 a 32x32 cell  $^3\text{He}$  gas multidetector will become operational which will increase the speed of data collection and will allow for an investigation of satellite or split peaks.



The new set-up of D9B providing a substantial flux gain and an increase of resolution at high scattering angles, compared with the former D9.

## D10, Four-circle triple-axis spectrometer

During the shut-down of the reactor the old Carine electronics were replaced by closed-loop microprocessor control modules and a multiplexor (designed by D. Bounolleau) to allow simultaneous positioning of the spectrometer angles.

The first cycle after the shut-down was devoted to tests and refinement of the new electronics and control software; the experimental programme was successfully started in the second cycle. In the four-circle triple-axis mode when up to six angles may be changed in each step the setting time decreased by as much as 50%.

The flux at D10 has increased by about 20% following the replacement of the guide tube at the position set free by D19A.



*A view of the experimental area around the H11 beam port. The monochromator turret in the middle is feeding 2 instruments: D2B, the very high resolution powder diffractometer (on the right) and D20 a new high flux multidetector diffractometer (in the foreground). A particular feature of the latter is its biological concrete shielding which has been designed such that it requires a minimum of space and yields a maximum of efficiency. The third instrument, the 2D Multidetector D19B is hidden behind the turret.*

### **D15, Four-circle MK6 diffractometer on the inclined thermal beam IH4**

D15 is one of the few instruments in the group which has not undergone any major modification during the long shut-down. It was available for experiments directly after the reactor start-up in September.

The first two weeks of the first cycle were used to test a small 32x32 cell position sensitive detector which it is hoped will be used on a regular basis at some time in the future. The object of installing this multidetector is both to improve the precision of normal integrated intensity measurements and to facilitate experiments on structures with incommensurate lattices.

### **D16, Four-circle MK6 diffractometer on cold guide H16**

No major changes have taken place on D16 during the long reactor shut-down. New software introduced now makes data

acquisition compatible with the other multidetector diffractometers at ILL. Data treatment programs have also been improved and extended. The automatic temperature controller for cryostats has been installed and design studies are underway for modifications to the monochromator-Be filter collimation configuration to improve both flux at the sample and resolution.

### **D19B, Multidetector diffractometer for protein crystallography on H11**

Following the dismantling, during the long shut-down, of D19A on the H24 guide, and successful tests of the new shielding on the H11 beam tube, the new instrument D19 was installed close to the old D8 position on the thermal beam H11. D19 has a choice of 'alpha-one' collimation and four take-off angles from 42° to 90° are available for reflection from focussing graphite, Ge or Cu monochromators. The 4° x 64° curved PSD already used on D19A has been re-housed in heavier shielding appropriate to the reactor environment. The new C-shaped Eulerian cradle, with offset phi-axis, will allow a much larger range in omega than was possible on D19A, and as well will accept a new compact 2-stage Displex cryorefrigerator which can cool to below 20K.

It is expected that agreement will be reached in early 1986 on the design of a new PSD to cover a solid angle at least five times that of the present detector. This would effectively increase the measuring efficiency by more than a factor of five, since with the present 'thin' 4° x 64° detector, edge effects are important.

D19 works most efficiently with large unit cells ( $a > 20\text{\AA}$ ), but will give much improved statistics even for single reflections because of its spatial resolution of  $0.125^\circ \times 0.25^\circ$ ; its use for samples other than proteins is not excluded.

### **D20, High flux multidetector diffractometer on thermal beam H11**

Following the successful tests of the new H11 beam tube protection, the instrument is being installed at its final position in the reactor. At the monochromator position (300 x 60 mm), the beam was found to be uniform with an intensity of about  $4 \cdot 10^9$  neutrons  $\text{cm}^{-2}\text{s}^{-1}$ .

The current version of D20 was designed to replace D2 in the field of high flux medium resolution powder and liquid diffraction.

It will operate with two fixed-curvature focussing monochromators: HOPG (2.5Å) and Cu-200 (1.35 and 0.87Å) and will be equipped with a 126 cell PSD.

### **DB21, Four-circle diffractometer with PSD for biological macromolecules on cold guide H15**

The instrument has entered the final stage of development during 1985. It was transferred to its permanent site on the H15 guide during the first half of 1985. A redesign of the monochromator housing and collimating elements was

necessary to reduce the gamma-radiation caused by the incoming beam itself, and to increase the incident neutron flux. The monochromator-sample distance is now 1m, giving a flux comparable to D17.

A prototype high-pressure helium detector (Jacobé) was tested on DB21 giving satisfactory results. The definitive one, with helium pressure of 8 atmospheres and a spatial resolution of 1.5 mm will be delivered in February 1986. The Anger-type scintillation detector with a resolution of 1.2 mm was delivered by Argonne National Laboratory and is being tested.

A new version of data reduction software was written and tested on the EMBL VAX 11/750, while the data acquisition software is being implemented on the instrument computer.

We expect the instrument to be available for scheduled experiments as from February 1986.

### S42 and S42B, Laue Diffractometers

On S42 the technique of the "White Beam" Laue diffraction was further developed. This method is advantageous for the **detection** of additional elastic diffraction spots of magnetic or nuclear origin (superlattice reflections) during phase transitions, for **evidencing** the symmetry change of the crystal and for the determination of the set of propagation vectors describing the modulated magnetic or nuclear structure.

The S42 instrument was duplicated in 1985 (S42B) to produce a standard D13-type instrument to orient and test the quality of single crystals. The potential of the 'White Beam' Laue diffraction technique will be developed with a greater variety of sample environment facilities than available on the original instrument.

Co-ordinator: J. Pannetier

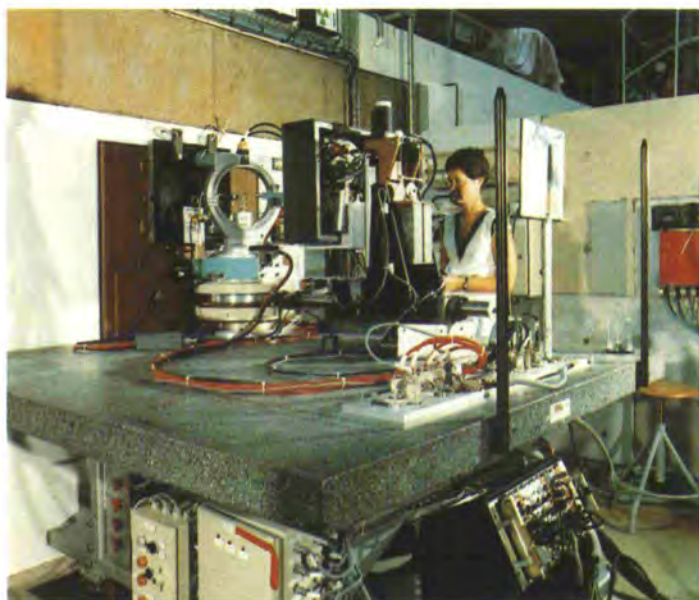
### S20, Neutron Topography Diffractometer

The small diffractometer S20, which is mainly devoted to neutron topography, has been moved to a new location where the flux is enhanced by more than a factor 3. This allows a reduction in the exposure times, which typically are now of the order of ~ 3 hours to obtain a spatial resolution on the topograph of ~ 60 microns.

It is expected that this improvement, as well as the new electronics, will make it easy for external groups to carry out in a reasonable time (~ 10 days) neutron topographic investigations of defects or domains on a given material (which usually implies to perform ~ 30 topographs).

### S21, Double Crystal Diffractometer

S21 is presently being reinstalled on the H25 thermal beam position. A flux gain of an order of magnitude and improved flexibility are expected from this move. The on-line Ir192 gamma-diffractometer tested last year will be installed on air cushions to facilitate the alignment procedure. User-operation will resume in spring 1986.



The picture shows the four-circle diffractometer with PSD for biological macromolecules DB21 at its final site on the H15 guide.

# SPECIAL STUDIES OF EXPERIMENTAL AND INSTRUMENTAL TECHNIQUES

(P. Ageron)

## EXPERIMENTS

### Check of the calculated performance of the modified vertical Cold Source:

The aims of the modifications on the vertical Cold Source were (i) to enhance the cold neutron flux in the direction of the existing horizontal cold neutron guides by means of a 'cavity' which penetrates at a depth of 25 cm into the 38 cm diameter sphere of the liquid deuterium moderator; (ii) to create a vertical beam of very cold and ultra-cold neutrons (VCN  $20 < \lambda < 200 \text{ \AA}$ , UCN  $\lambda > 600 \text{ \AA}$ ) by means of a guide penetrating into the liquid deuterium moderator.

The measurements carried out in cooperation with the pertinent instrument responsables, the Health Physics Group and the Reactor Department have confirmed more precisely the calculated predictions (reported in 'Neutron Scattering in the Nineties, January 1985, IAEA CN 46/16):

The flux in the horizontal guides has been increased by a factor 1.6 - 1.7 for neutron wavelengths between 3 - 6  $\text{\AA}$  (instruments D7 and IN10 on H15). The gain is smaller at longer wavelengths, as indicated by measurements on D11 and by activation of gold detectors (integrated gain of about 1.2 - 1.4).

The overall gain factor for VCN (TOF measurements by A. Steyerl and gold foil measurements) and UCN (storage measurements, in a 'standard' bottle, by W. Mampe and A. Beynet) of the vertical beam (before the installation of the turbine) compared with the guide PN5 aiming at a room temperature moderator, is about 75. This figure exceeds the calculated (with large uncertainties) cold moderator effect and includes a better reflectivity of the vertical guide. A more detailed description of the whole facility is given in the section: Instrument Group 'Nuclear and Fundamental Instruments'.

### Operation of a goniometer on PN5, dedicated to VCN:

Its modular design includes independent  $\theta$  and  $2\theta$  scanning, TOF option, possibly on a horizontal sample position (surfaces of liquids). Its first purpose is the precise determination of reflectivity profiles of surfaces by means of VCN (20 - 100  $\text{\AA}$ ). Measurements have been made or are in progress on the qualification of metallic mirrors, in particular for the in-pile guides of the second cold source (K. Gobrecht) and for the development of multilayer monochromators (W. Kaiser).

## CALCULATIONS

Among the various studies made by means of programs written by A. Robert, one should mention:

- determination of the effectiveness of  $^{58}\text{Ni}$  instead of natural Nickel as a coating for the guides of the second cold source. With Monte Carlo simulation, gain factors from 1.4 - 1.5 at 3  $\text{\AA}$  to 1.2 - 1.3 at 11  $\text{\AA}$  have been calculated.
- optimization of monolayer ( $^{58}\text{Ni}$ ) thickness and of multilayers (number, spacing, composition) (in progress).
- simulation of the trajectories, including gravity effects, of UCN in parallelepiped bottles, which permit the determination of three UCN loss rates, on the walls, in real conditions, without *a priori* assumption of a density homogeneity. It will be used in connection with a scheduled experiment on the determination of the neutron life-time.

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**DIRECTORATE DEPARTMENT**

# SCIENTIFIC COORDINATION AND PUBLIC RELATIONS

## SCIENTIFIC PROGRAMME IN 1985

Following the successful completion of the reactor renewal programme a quasi normal resumption of the scientific programme began on 3 September 1985. It will be recalled that, as well as the work uniquely associated with the reactor, the Institut had planned an intensive programme of instrument installation, modification and modernisation which was to take place, where possible, during the reactor shut-down. Some aspects of this programme were inexorably linked to the main reactor planning and, for example, certain primary spectrometers could not be installed before completion of the associated beam tube replacement. This resulted in both, a heavy work load during the last few weeks of the reactor shut-down for the engineers and technicians responsible for installation, and a limited availability of instruments for the scientific programme when the reactor went to full power. In addition, heavily modified instruments required neutron tests before being handed over to external users, again delaying availability during the first reactor cycle.

Such perturbations as occurred were, in general, expected and had been taken into account during the planning stages of the scientific programme which was to follow the reactor shut-down. It was also expected that with such a protracted shut-down at the world's foremost neutron scientific laboratory, interest in the resumption of operations would be intense and in fact the Secretariat received 643 applications in February 1985 requesting, in total, 7502 instrument/days. For various reasons, mainly oversubscription, the final planning required precise dates for 403 experiments and it is to the credit of all concerned with the reactor overhaul and instrument installation that, despite the advanced nature of the nuclear and scientific engineering, they were able to say some months ahead the exact date for a full resumption of the scientific activity at the Institut.

The scientific programme was accordingly resumed on 3rd September 1985 and the first few days were reserved for neutron tests in order to re-commission instruments which had stood idle for 11 months and also to test modifications which had taken place during this period. Other relevant changes which had been in the shut-down programme, such as the replacement of the vertical cold source and in-pile neutron guides gave a cold neutron flux increase of some 65%, dependent on wavelength, and had to be included in the instrument operational procedures. However, the consequences of these initial perturbations on the instrument availability were not particularly profound and after two weeks following the reactor start-up the scientific programme was continuing normally and remained so until the end of the report year.

Evidently, a statistical review of the scientific programme for 1985 is not significant after such a short operational period and will not be included in this report. However, a full account will be taken of this session in the instrument review of 1986.

## WORKSHOPS AND CONFERENCES

The Office has organized and assisted in organising the following conferences in 1985:

- 1) 'Diffusive Molecular Motion', 4-9 March 1985 (Responsibles: J.-P. Beaufils, M. Bée)
- 2) 'Interpretations and Uses of Electronic Distribution', 26-29 March 1985 (Responsibles: P.J. Becker, P.J. Brown, F.L. Hirschfeld)
- 3) 3rd International Conference on the 'Structure of Non-crystalline Materials', 8-12 July 1985 (Responsibles: C. Janot, A. Wright)
- 4) 'Investigations of Higher Order Correlation Functions', 15-16 July 1985 (Responsible: J.B. Suck)
- 5) 'Evaluation of Data from PSDs', 4-8 November 1985 (Responsible: M. Roth)

The workshops to be organized in 1986 are:

- 1) 'Structure and Dynamics of Interfaces', 7-8 April 1986 (Responsibles: J.-P. Beaufils, D. Richter)
- 2) 'Quantum Motions in Condensed Matter', 24-26 September 1986 (Responsibles: A. Heidemann, A. Magerl, D. Richter, (ILL), M. Prager, T. Springer (Jülich))
- 3) 'Fission in 1986', Autumn 1986 (Responsible: H. Faust)

## LIBRARY

1985 BUDGET: 714 KF

The Library now has 8600 books, 7700 reports and handles 290 subscriptions to journals of which 100 are dispatched to other departments.

- Journal subscriptions were still the main expense and represented 65% of the budget (global increase of 20% between 1984 and 1985)

- 446 volumes were bound

- Book and serial acquisition remained almost steady (400 books of which 90 were deposited with the departments)

- Purchasing of reprints was kept at a very low level, as for 1984.

In 1985, the Library was directly connected to Transpac which allowed direct access to computerised data bases and on-line ordering of documents (both applications increasing in number).

- Due to the long reactor shutdown, the number of experimental reports collected was very low.

- 300 publications with I.L.L. authors were registered which brings to 3000 the computerized references searchable by author, instruments, college related to ILL measurements.

## SAFETY AND HEALTH PHYSICS GROUP

In the Health Physics field, 1985 was particularly characterized by the monitoring of work carried out during the long shut-down.

The Safety and Health Physics Group was closely associated with the maintenance work on the reactor as regards the definition of working conditions, decontamination and waste disposal. Highly active parts, such as the mainly zircaloy Cold Source and the reflector tank 'chimney' (low-cobalt stainless steel) were removed, together with particularly large and active items such as the H1-H2 beam tube thimbles.

The Group was also associated with the definition of radiological shielding, especially on the new instrument beam tubes H11 and H3.

Routine control work has continued:

- Dosimetry of ILL staff and visitors.
- Monitoring and control of radioactive sources used for experiments or for instrument calibration.
- Inspection of X-ray equipment.
- Monitoring of radioactive liquid and gaseous waste.

Specific studies have been carried out on the use of certain equipment, such as choppers and cryomagnets, and certain targets, including transuranic elements.

The study of the type of equipment necessary for the replacement of the reactor health physics monitoring was carried out in collaboration with the reactor department.

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**INSTRUMENTS AND METHODS DEPARTMENT**

## EINLEITUNG

Das Jahr 1985 war hauptsächlich gekennzeichnet durch den langen Stillstand für Erneuerungsarbeiten am Reaktor, der für die Abteilung Instrumente und Methoden zahlreiche Aufgaben mit sich brachte.

Neben der den Reaktorgruppen geleisteten Unterstützung, und zwar - durch die Mechanik-Werkstatt (dringende oder spezielle Bearbeitung von aktivierten oder kontaminierten Teilen)

- oder durch die Versuchshalle (vollständige Justierung der Neutronenleiter H1-H2)

ist der grösste Teil der Instrumente der Experimentierhalle des Reaktors abgebaut, meistens geändert und verbessert, und schliesslich zum Wiederanfahren des Reaktors Anfang September 1985 wieder aufgebaut worden.

Ebenfalls anlässlich dieses langen Betriebsstillstands sind grundlegende Änderungen vorgenommen worden:

- an D9, dessen Monochromator näher an die Neutronenquelle gebracht wurde,

- an D11, dessen Multidetektor im Innern des Flugrohres beweglich angeordnet wurde, um eine beträchtliche Zeiteinsparung bei der Verfügungstellung des Instruments zu erlauben.

So ist auch die neue Ausrüstung des Strahlrohres H11 mit einer speziellen Abschirmung fertiggestellt worden, die die Öffnung von 3 voll-

## INTRODUCTION

The year 1985 was mainly characterized by the long shutdown for reactor renovation, which had numerous consequences for the Instruments and Methods Department.

Apart from the assistance given to the teams in the Reactor Department

- by the mechanical workshop (urgent machining or special work on activated or contaminated parts)

- and by the testing workshops (complete realignment of the guides H1-H2), the majority of the instruments in the reactor experimental hall were dismantled, most of them modified and improved, then re-assembled for the start-up at the beginning of September 1985.

During this long reactor shutdown, fundamental modifications were carried out on

- D9, where the monochromator was brought closer to the neutron source,

- D11, where the multidetector was made mobile inside the flight tube, permitting a considerable saving on the time for making the instrument available.

Similarly, the re-equipment of beam tube H11 was completed with

## INTRODUCTION

L'année 1985 a été marquée essentiellement par l'arrêt de longue durée pour renovation du réacteur, dont les conséquences ont été multiples pour le Département Instruments et Méthodes.

Outre l'aide apportée aux équipes du département Réacteur,

- par l'atelier de mécanique (usinages urgents ou spéciaux sur des pièces activées ou contaminées);

- ou par le Hall d'Essais (réalignement complet des nappes de guides H1-H2), la plus grande partie des instruments situés au niveau C du Hall Réacteur ont été démontés, la plupart du temps modifiés et améliorés, puis remontés en vue du démarrage qui a eu lieu début septembre 1985.

Toujours à l'occasion de ce grand arrêt de l'exploitation, des modifications fondamentales ont été apportées : à **D9** dont le monochromateur a été rapproché de la source de neutrons, à **D11** dont le multidetecteur a été rendu mobile à l'intérieur même du tube de vol permettant un gain considérable sur le temps de mise à disposition de l'instrument.

De même le nouvel aménagement du canal H11 a été mené à son terme avec une protection spéciale permettant l'ouverture de 3 faisceaux totalement indépendants pour les nouveaux instruments D19, D20, D2B.

kommen unabhängigen Strahlenbündeln für die neuen Instrumente D19, D20 und D2B erlaubt.

Ein in die neue vertikale kalte Quelle eingebautes Strahlrohr für ultrakalte Neutronen hat zur Installierung von S-Instrumenten von sehr grossem wissenschaftlichem Interesse geführt.

Im Bereich der Messmethoden mit Neutronen sind die Bemühungen bezüglich der Herstellung von Berylliummonochromatoren mit dem MPI Stuttgart erfolgreich fortgesetzt worden, während ein mit Carbone Lorraine geschlossener Vertrag es gestattete, am ILL die Studien bezüglich einer Pressofens für die Herstellung von mosaikförmigen Graphitkristallen zu beginnen.

Die Beherrschung der Gasmultidetektortechnologie ist vertieft worden, und zwar durch eine systematische Analyse einer grossen Anzahl von möglichen Gasmischungen und durch die Anwendung der Technologie der gedruckten Schaltungen mit dem Ziel, die Drähte durch auf Glas aufgetragene metallische Strukturen zu ersetzen.

Der Bau des künftigen für D20 vorgesehenen, aus 1600 Zellen bestehenden Bananendetektors ist in Angriff genommen worden.

Was die Organisation der Abteilung anbetrifft, so ist die Elektronikgruppe einheitlicher gestaltet worden, wodurch dem wissenschaftlichen Bereich eine immer raschere und wirksamere technische Unterstützung geboten werden kann. Ausserdem wird hiermit eine Grundlage für die bessere Standardisierung der Lösungen für die die Steuerung moderner Instrumente gestellten Aufgaben geschaffen.

a special shielding permitting the opening of three completely independent beams for the new instruments D19, D20, D2B.

An ultra-cold neutron tube installed on the new vertical cold source permitted the installation of special instruments of great scientific interest.

In the field of measuring methods with neutrons, efficient collaboration with the MPI Stuttgart continued for the production of beryllium monochromators, while a contract signed with Carbon Lorraine enabled a design study to start at ILL on a hot press for the production of graphite mosaic crystals.

Knowledge of the gas multidetector technology was improved by a systematic analysis of a large number of possible gas mixtures and by the application of circuit technology for the replacement in due course of wires by metallic structures deposited on glass.

Construction of the future 1600-cell "banana" on D20 has been started.

Finally as regards organization a more homogeneous electronics group has been set up, making it possible to give the scientific sector an ever more rapid and efficient technical support, while establishing the basis of a better standardization of the solutions to the problems raised by modern instrument control.

Un tube de Neutrons Ultrafroids installé sur la nouvelle source froide verticale a conduit à la mise en place d'instruments spéciaux d'un très grand intérêt scientifique.

Dans le domaine des méthodes de mesure avec neutrons, les efforts se sont poursuivis efficacement avec le MPI de Stuttgart pour la production de monochromateurs de Béryllium, tandis qu'un contrat signé avec le Carbone Lorraine permettait le début de l'étude à l'ILL d'un four de pressage pour la production de cristaux mosaïques de Graphite.

La maîtrise de la technologie des multidetecteurs à gaz a été approfondie par une analyse systématique d'un grand nombre de mélanges gazeux possibles et par l'application de la technologie des circuits imprimés afin de remplacer à terme les fils par des structures métalliques déposées sur verre.

La construction de la future banane 1600 cellules destinées à D20 a été lancée.

Enfin sur le plan de l'organisation, un service électronique plus homogène a été mis en place, permettant d'offrir au secteur scientifique un support technique toujours plus rapide et efficace tout en jetant les bases d'une meilleure standardisation des solutions aux problèmes posés par le contrôle d'instruments modernes.

# 1. MECHANICAL CONSTRUCTION

(Gobert - Blum)

The work of the group is in four sectors:

- Design studies
- Subcontracting
- Production workshop
- Assembly, tests and measurements.

## Design studies

The continued equipment of new instruments resulted in design studies on D19B, D2B, D20, IN20, IN1, D4. There were also additional studies in connexion with the completion of D9B and D11C. These last two instruments were tested at the reactor re-start.

Modifications were designed for IN4, IN8, IN11, D3 and D5, and the design work has been carried out and the invitation to tender issued for the shutter on the horizontal cold source neutron guides for the reactor department.

Design studies also started this year on IN14.

## Construction work

The subcontracted work represents 215 jobs (in comparison with 192 in 1984). These cover experimental instruments and requests from the instrument operation and reactor departments.

The budget involved was 1934 KF.

As regards development work, reference should be made to the choppers, encoder modules and heavy duty goniometers, on air cushions and designed for high magnetic fields (superconducting magnets).

## Assembly, tests and measurements

After preliminary assembly in the test workshops, the newspectrometers D9, D19 and D20 and their electrical equipment were installed in position.

All the staff have been greatly involved in the work of the long shutdown. In particular the dismantling and reconstruction including realignment of the neutron guides H1-H2 were entirely the responsibility of this group. Most of the experimental instruments were the subject of maintenance work. Particular reference should be made to the complete modernization and partial rebuild of D5 and IN11 and the selectors and choppers on D7, D11, IN10, IN13, D17 and IN11.

Numerous dynamic measurements (vibration, constraints, temperature, flow, sound level) have been carried out for the Instrument Operation and Reactor Departments. These represented 20% of the working time.

New materials have been tried, for application to chopper discs. Thus D7 and IN13 use permaglass discs impregnated with gadolinium oxide. A test disc has operated up to a peripheral speed of 497 m/second.

On the multichopper instrument IN5, which has recently received KFA Jülich type passive magnetic bearings, this group has installed the measuring and safety equipment.

In the electro-technical field, the automation and safety equipment for the new instruments D2B, D9, D11, D20, D17 (re-built) has been developed, from the design stage to the tests on site.

The team responsible for mechanical metrology has continued its activities as regards inspection, testing and development of operational parts and assemblies, and this year particularly on the planar monochromators and analyzers on instruments such as IN1, D9, D2B, D20, IN20 (focussing system for polarized neutrons).

# 2. ELECTRONICS GROUP

(R. Klesse)

In addition to a variety of intervention work on instruments listed below, the following lines of action may be distinguished. One cadre has been allocated to electronic developments requiring increasingly the use of microprocessors by the introduction of the VME-Bus associated with the Motorola 68000 microprocessor. This new "standard" is in addition to the CAMAC, widely used at ILL, that will only replace it in cases where the expected performances require (time of flight electronics, rapid display of data during acquisition, phasing of choppers, etc.). This is in line with the current transition for computing staff between PDP11 series minicomputers and Micro-VAX micros.

The introduction of the new bus is being accompanied by a coordinated training effort for technicians on micro-programming.

The existing CAMAC features are gradually being generally introduced in order to facilitate maintenance. A considerable number of movements on ILL instruments have been newly equipped with a closed loop controller, permitting the use of a DC motor.

In more detail, the following work has been carried out or started in 1985.

Gradual introduction of the CAMAC scales on the triple axis instruments as SOLARS are replaced by PDP11/73. This modification has been completed on IN8 and has now been started on IN1 and IN3. Final installation of IN20.

D2B: Transfer of the instrument from the guide hall to the H11 position and verification of three additional axes (new monochromator).

D4B: Replacement of the MACAMAC by a PDP11/23 with CAMAC DEC controller.

D9, D10 and D1A: Replacement of the CARINE electronics by 28 closed loops.

D11: a) Motorization of the detector carriage, the beam stop (x + y) and the sample changer with 4 closed loops.

b) Data transfer by optical line

D17: Change of encoders: re-cabling / motorization of the beam stop, detector and sample changer - 4 closed loops.

D19B: Transfer of D19 from the guide hall to the H11 position, motorization of 11 axes, including 5 with closed loop and 6 on manual.

D20: Commissioning of the "multiscaling" system. Installation of the instrument at the H11 position.

IN4: Contribution to installation of choppers on magnetic bearings and development of safety electronics to ensure permanent phasing and to make any asynchronous operation impossible (protection of motor).

IN5: a) Contribution to the installation of choppers on magnetic bearings;  
b) Commissioning of a programmable time of flight unit.

PN4: Reconstruction around a PDP11 with CAMAC controller incorporating GAMS, which was already equipped with a CAMAC acquisition system.

## 3. MONOCHROMATORS

### (A. Freund, B. Hamelin)

D13A : Neutron double-crystal diffractometer on thermal guide H24

D13C : Neutron double-crystal instrument on thermal guide H23

Li2A : X-ray double-crystal diffractometer

Li2B : X-ray orientation unit

Li3A-F : Gamma-ray diffractometers

Laboratory for crystal preparation

### STATUS OF THE INSTRUMENTS

D13A served as usual for crystal alignments and short experiments, mostly neutron topography. The flux on this instrument was decreased by a factor 2 because the instrument S20 (topography) was moved to the former D13B position upstream (before) D13A.

D13C operated at the CENG's SILOË reactor until the restart of the ILL HFR. Its computer control is being replaced by a new microcomputer which will permit a more easy and rapid execution of crystal orientations and tests urgently needed in view of the high workload for the present monochromator production.

Li 2A was completely reconstructed and performs satisfactorily after some initial troubles with the air-cushion technique. Beryllium crystals are being tested routinely using diffraction profile analysis and X-ray topography. The same rotational and translational stages are available as at D13C and will be used for Be monochromator assembling.

Li 3 worked routinely, supplying  $\gamma$ -radiation from two sources of 0.0301 and 0.0187 Å wavelength to five individual diffractometers for carrying out experiments; low and high resolution analysis of crystal perfection, in-beam plastic deformation at high temperatures and in-beam growth of crystals for neutron monochromators. By the end of 1985 about 80 (6 cm in diameter and 15 cm long) were grown or re-grown in cases where the subsequent plastic deformation had failed to give satisfactory results. More than 60% of these crystals were of sufficient quality to be further processed for monochromators.

## MONOCHROMATOR PRODUCTION AND DEVELOPMENT

Only a fraction of this work can be considered as 'routine' and still a large part needs a careful examination to find the best solution for each individual monochromator requirement. This is also one of the reasons why complete focussing monochromators are not commercially available. Many neutron scattering laboratories were advised by the ILL or even used the ILL facilities when designing and constructing their monochromators. Of course, care was taken to organise these activities such that the ongoing monochromator preparation for ILL instruments is not disturbed. This is proved by the achievements in 1985 which are shown in the following table:

Instrument	Material	Reflection	Dimensions (mm)
D2B	Ge	511	50 x 300
D9B	Cu	220	70 x 70
D19	Ge	311 - 511	100 x 120
IN3	Cu	111	150 x 75
IN8	Cu	111	160 x 150
IN8	Cu	220	160 x 160
IN20	Cu	111	120 x 150
IN20	Cu MnAl	111	75 x 75
Under preparation:			
D19B	Cu	220	100 x 120
D20	Cu	200	80 x 300
IN20	Cu	220	120 x 150

These monochromators are composite focussing devices (except the one for D9B). One has to add the testing and assembling of many pyrolytic graphite pieces to build monochromators or analysers for the instruments D1B, D19B, IN1B and IN20. The total of 12 monochromators completed in 1985 represents an absolute record even when taking into account that part of the material needed had been prepared in 1984.

The joint project for the production of Be monochromators went on smoothly on the existing facilities at the Pulvermetallurgisches Laboratorium of the Max-Planck-Institut für Metallforschung at Stuttgart. More than 30 crystals with average dimensions of 14 mm diameter and 70 mm length were grown and proved all to be of good quality : single-peak rocking curves of about 4 minutes width over a useful length of at least 50 mm. Some of these crystals are currently deformed under tensile stress which is easier to perform and should give similar results compared to the plastic deformation under compression which had been planned originally for producing the desired mosaic spread. The construction of the new facilities, i.e. the triple ellipsoid light furnace and an induction heating facility for materials purification prior to crystal growth, was delayed due to late delivery of several parts. It is hoped that both machines will be operating by the end of 1985.

In view of the extremely high cost of top quality pyrolytic graphite and encouraged by an excellent result obtained at the laboratory of the firm Le Carbone Lorraine, Gennevilliers, it was decided to start the development of PG monochromators at the ILL, with the participation of Le Carbone Lorraine.

The design of the induction heated press (3000°C, 50 tons) including the in-beam control of the mosaic spread with  $\gamma$ -ray diffractometry is almost finalised. The project is the subject of a thesis and the facility will be installed at the Laboratoire Louis NEEL of the CNRS-Grenoble.

## 4. POSITION SENSITIVE DETECTOR DEVELOPMENT AND CONSTRUCTION

(A. Oed, J. Jacobé)

### DEVELOPMENTS

A new vacuum system with two different chambers for the testing of gas detectors has been constructed.

The first part was operational in Summer 1985. Thus the physical properties as to the use as detector medium of a dozen gases and gas mixtures have so far been measured. Part two of the device will be ready at the end of the year. It consists of a neutron detector which can easily be disassembled and which is built in standard UHV technique. This part will give the opportunity of testing thoroughly new arrangements for the detector electrodes and of applying the technical findings to the construction of detectors with large dimensions.

On the one hand, the traditional arrangements using wires for positioning by local charge avalanches should be simplified as far as possible in order to permit a partially automatic production, especially for curved detectors such as the so-called bananas.

On the other hand an effort has been made to eliminate completely the wires, which are the main source of many technical and electrical problems. They will be replaced by a structure of conductors applied to a glass substratum. The technical problems of producing such an electrode have almost been solved since the procedures already used for the fabrication of integrated circuits were adopted. First preliminary experiments to test this point are encouraging.

'Foil' detectors in which the neutrons are captured in a thin foil of high absorption cross-section and where the escaped ionizing particle delivers the signal have the advantage of small parallax errors. Tests with the Gd-detector for DB 21, however, have shown that a certain background level produced by  $\gamma$ -s of the environment is unavoidable. A simple calculation shows that the detection probability of Li is comparable to the detection probability of natural Gadolinium and has the additional advantage of easy discrimination of the background. A lithium detector, similar to a parallel avalanche counter, was therefore developed and will be tested at the end of the year.

Finally a LiI scintillator is now available. A neutron detector counting the light pulses by means of solid state photodiodes will be investigated.

## MAJOR PROJECTS

### (A) Modifications of detectors and/or electronics

D11: Detector replacement and associated new electronics. Start of recabling of replacement detector.

D19: Complete overhaul of the multidetector before its installation at position H11 close to the reactor.

D20: Improvement of the 128-cell prototype. Design study for 1600-cell detector started.

D4: Construction of a third multidetector by modification of the former D2.

### (B) Miscellaneous

Manufacture of special monitors (uranium) for instruments D4 and D20.

Development of new 32-channel cascable cards for the final detector on D20.

General overhaul of all instruments during the reactor shutdown.

Miscellaneous filling of monitors and detectors for instruments IN13, PN5, etc.

Estimate for electronics supplies 400: TOF detectors and a x-y detector for HMI.

D15: Construction and commissioning in September of a 32 x 32 cell multidetector with 2 mm steps.

D9: Manufacture in progress of an identical detector with a lighter entry window (zirconium + aluminium).

DB21: Construction of a final 128 x 128 wire detector with 1.5 mm steps with positioning by internal delay lines.

Acceptance inspection of an Anger Camera type scintillation detector produced by Argonne.

## 5. MULTILAYER LABORATORY

22/119 PH22 (FOH, 7181) D117 04/253  
 (O. Schärpf, W. Kaiser, PH4 04/343  
 W. Just, W. Graf)

The production of supermirrors for the assembly of polarizers continued on the RIBER and ELECTROTECH evaporators without major problems. The quality testing of the supermirrors was carried out at the Institut für Reaktortechnik at Würenlingen (Switzerland) during the shutdown of the ILL reactor.

The completed polarizers, the value of which amounts to 570 KF in 1985, are intended for the ILL (D7, D1B, IN12) and for external laboratories.

New developments include polarizers with supermirror deposits on both faces and focussing supermirrors for neutron guides. A further development area is that of multilayer monochromators for ultracold neutrons. The double layers of aluminium-titanium, aluminium-nickel or nickel-titanium with a thickness of 90 to 200 Å and with 40 to 400 Å or more of double layers were deposited by evaporation on ILL equipment or by spraying elsewhere (BNL and BALZERS). Comparative measurements with cold neutrons of 7.2 Å on S3 and with ultracold neutrons of 15 to 60 Å on SN5 are in progress.

## 6. SPECIAL INSTRUMENTS

(W. Drexel)

In the field of 'S'-activities the long reactor shut-down was fully utilized for major modifications and the installation of new experimental equipment, especially in the field of ultracold neutrons. The most important activities are summarized as follows:

On **S18** (neutron interferometer) the motor drive modules were improved. The instrument, however, still suffers from frequent break-downs; a program has been set up to cure this situation by early 1986.

**S20** (neutron topography) was moved somewhat forward (in front of D13A) in order to free space for S42. Its electronic control system (1984) based on a MACAMAC and BASIC language has received the necessary software and is now giving full satisfaction.

**S21** (high resolution double crystal spectrometer) was moved to position H25C where S6 was previously installed; it will gain about one order of magnitude in intensity. Furthermore, S21 received a new computer (PDP 11/23+) and new software.

**S34** ( $\gamma\gamma$  coincidence instrument) received a DEC CAMAC-controller for standard data incrementation into external memories as well as a new software system.

**S42** (the Laue diffractometer) is being rebuilt on the basis of a proposal by Baruchel. It has acquired commercially available mechanical modules; a cryostat (from D5) and an electromagnet (from D1B) will fit on this instrument in the future.

**S50** (the  $h/m$  precision measurement) obtained an improved, stronger supporting structure; the delivery of the definitive apparatus being slightly delayed, the original test set up has been reinstalled on this beam.

**S52** (a neutron life time measurement) acquired a PDP 11/23+ with the RSX11M operating system as well as most of the necessary data acquisition software. The background in this experimental area has to be reduced and a new shielding set up has been designed.

For **SN5** (the UCN source at the inclined beam hole on level C) a Canberra multichannel analyzer was linked to an HP personal computer for more sophisticated data treatment applications.

**SN7** (the polarized cold neutron beam) was provided with an air-conditioned cabin to house the electronic equipment for instruments to be installed on this beam. For the moment PERKEO, studying the neutron decay parameters, has been reinstalled there.

The neutron electric charge experiment has been reinstalled on the H18 beam with an improved neutron optical set up.

The most important activity in the field of 'S'-instruments in 1985 was certainly the implementation of the new source for very cold and ultracold neutrons on the upper floor of the reactor building. Originally proposed by P. AGERON (to install a vertical guide tube in combination with a neutron turbine it was decided in 1981 that this project should become an S-activity with A. STEYERL from the Technische Universität at Munich, where the necessary technology had been developed, as project leader. The finance was approved by the

German BMFT. In this new source a vertical metallic neutron guide (Ni) - consisting of a lower straight section of 5 m length and an upper curved section of 12.8 m length and 13 m radius of curvature - dips its lower end into ILL's vertical liquid deuterium cold source and conducts neutrons with wavelengths greater than 20 Å to level D. One half of the beam (7 x 3.5 cm) supplies very cold neutrons (centred around 70 Å) to the new experimental area; the other half of the beam is Doppler-shifted into the UCN range ( $\lambda > 200$  Å;  $v < 15$  m/s;  $E < 1.2$  μeV) by total reflection of the neutrons on a total of 690 receding Ni blades (160 x 150 mm<sup>2</sup>), 30 m/s, radius of curvature 65 mm) mounted on the periphery of the neutron turbine wheel ( $r = 800$  mm,  $v = 300$  rpm).

The UCN exit of the turbine will be equipped with a beam distributor guiding the full main UCN beam to only one instrument at a time, it can serve a maximum of 3 instruments on a time-sharing basis.

In order to fully utilize the UCN-beam another two side beams will be extracted mainly for test and development purposes.

Up to now the neutron guide tube, the vessel housing the neutron turbine as well as the pumping and vacuum control systems have been installed. The turbine itself was mounted at the end of November 1985.

The platforms - necessary for most of the planned experiments due to the fact that the neutron beam level is about 3 m above the floor - became available as from December 1985. The first experiments (UCN : neutron microscope; VCN : magnetic neutron bottle) will begin in December 85/January 86. 1986 will be the year of the start of the scientific exploitation of this unique new VCN-UCN source. Intensity measurements show that the gain factor over SN5 is about 75 and about 2000 compared with the original vertical guide tube at the Garching Reactor.

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**REACTOR OPERATION DEPARTMENT**

## **EINLEITUNG**

Das nach 13 Jahren ununterbrochenen Betriebs im Oktober 1984 begonnene Programm der Reaktorrenovierung ist während des ersten halben Jahres 1985 fortgesetzt worden.

Dieses Programm umfasste insbesondere den Austausch der am meisten bestrahlten Teile des Reaktors sowie die Reparatur des Schwerwasserkollektors im Reaktortank und den Ersatz der bestehenden vertikalen kalten Quelle durch ein neues leistungsfähigeres Modell.

Die Monate Juli und August sind den verschiedenen Wiederaufbauten und Versuchen gewidmet worden, die es erlaubt haben, die Genehmigung zum Wiederaufstart des Reaktors Ende August zu erhalten. Seitdem ist der gewöhnliche Rhythmus der 44-tägigen Betriebszyklen wiederaufgenommen worden. Seit dem Wiederaufstart sind zwei vollständige Zyklen durchgeführt und ein dritter Zyklus begonnen worden.

## **INTRODUCTION**

The reactor renewal programme, which started in October 1984 after 13 years of continuous operation, continued for all the first half of 1985.

This programme included in particular the replacement of the most irradiated parts of the reactor, the repair of the heavy water collector in the reflector tank and the replacement of the existing vertical cold source by a new, more powerful model.

The months of July and August were devoted to the various re-installations and tests which made it possible to obtain the authorisation to restart the reactor at the end of August. The usual rhythm of 44-day cycles of reactor operation has since resumed. After the restart two complete cycles were effected and a third cycle started.

## **INTRODUCTION**

Le programme de rénovation du réacteur après 13 ans de fonctionnement continu, commencé depuis le mois d'octobre 1984 s'est poursuivi tout au long du 1er semestre de l'année 1985.

Ce programme comportait notamment le remplacement des parties les plus irradiées du Réacteur, ainsi que la réparation du collecteur de reprise de l'eau lourde dans le bidon réflecteur et le remplacement de la Source Froide Verticale existante par un nouveau modèle plus performant.

Les mois de juillet et d'août ont été consacrés aux différents remontages et essais qui ont permis d'obtenir l'autorisation de redémarrage du réacteur à la fin du mois d'août. Depuis le rythme habituel des cycles de 44 jours de fonctionnement a repris. Après le redémarrage deux cycles complets ont été effectués, et un troisième cycle commencé.

# REACTOR OPERATION 1985

Tests and operation of the reactor at varying power between 26 August and 3 September.



The Director, Prof. Haensel (in the middle), with members of the reactor staff closely watched the start-up of the HFR on 26 August 1985.

## Cycle 1/85

Operation from 3 September to 17 October. The scheduled dates were respected and the cycle was completed without incident. Power was reduced during the last six days of the reactor cycle to compensate for the consumption of the fuel element during the tests preceding the normal cycle.

## Cycle 2/85

Operation from 29 October to 12 December. The scheduled dates were respected. The start of the cycle was interrupted by numerous short shut-downs (5), due in particular to safety rod n° two releasing; these shut-downs never exceeded half an hour.

## Cycle 3/85

The cycle started on 17 and 18 December, but was then interrupted to repair a heavy water leak on the control rod and for the Christmas holiday period.

## Data for 1985

No. of days originally scheduled :	95
actual No. of days of operation :	89.5
No. of equivalent days of full power :	85.3
actual operating time as proportion of year :	25%
actual operating time in relation to time :	94% scheduled
No. of fuel elements used :	3
No. of fuel elements despatched for reprocessing :	4
No. of unscheduled shut-downs :	11
including: brief shut-downs :	10
shut-downs with Xenon poisoning :	1

# WORK OF THE VARIOUS GROUPS

## REACTOR BLOCK

After completion of dismantling of the beam tube thimbles which started at the end of 1984 for H12, H6/H7, IH3, IH1 and finally H1/H2, the cutting of this last beam tube thimble was started; the large size of the beam tube thimble H1/H2 meant that it could not be removed in a transport container, and this active part had to be cut into several sections underwater in the swimming pool adjacent to the central core; the machine developed for this cutting work with a diamond wire was designed by the Reactor Department drawing office. It was built in close collaboration between industry and the Reactor Department.

At the beginning of 1985 this operation was followed by the development and tests of a second spark erosion machine which permitted the cutting-off by remote control of the damaged part of the heavy water collector in the reflector tank. On completion of this work, the new beam tube thimbles were installed, as was the new ultra-cold neutron loop in beam tube IH3. The new vertical cold source (SFV3) was installed with the new tube H1/H2.

Associated with this new cold source, a vertical guide tube and a curved guide tube were placed in the reactor swimming pool to permit a new ultra-cold beam tube to be brought out in the upper level of the reactor hall.

The verification and re-installation of the components of the reactor block (safety rods, control rod, natural convection valves, anti-siphonage valve, 'chimney', fuel element support, reactor valve, neutron guides for beam tube H1/H2, etc) were followed by tests on the various circuits which preceded the restart of the reactor.

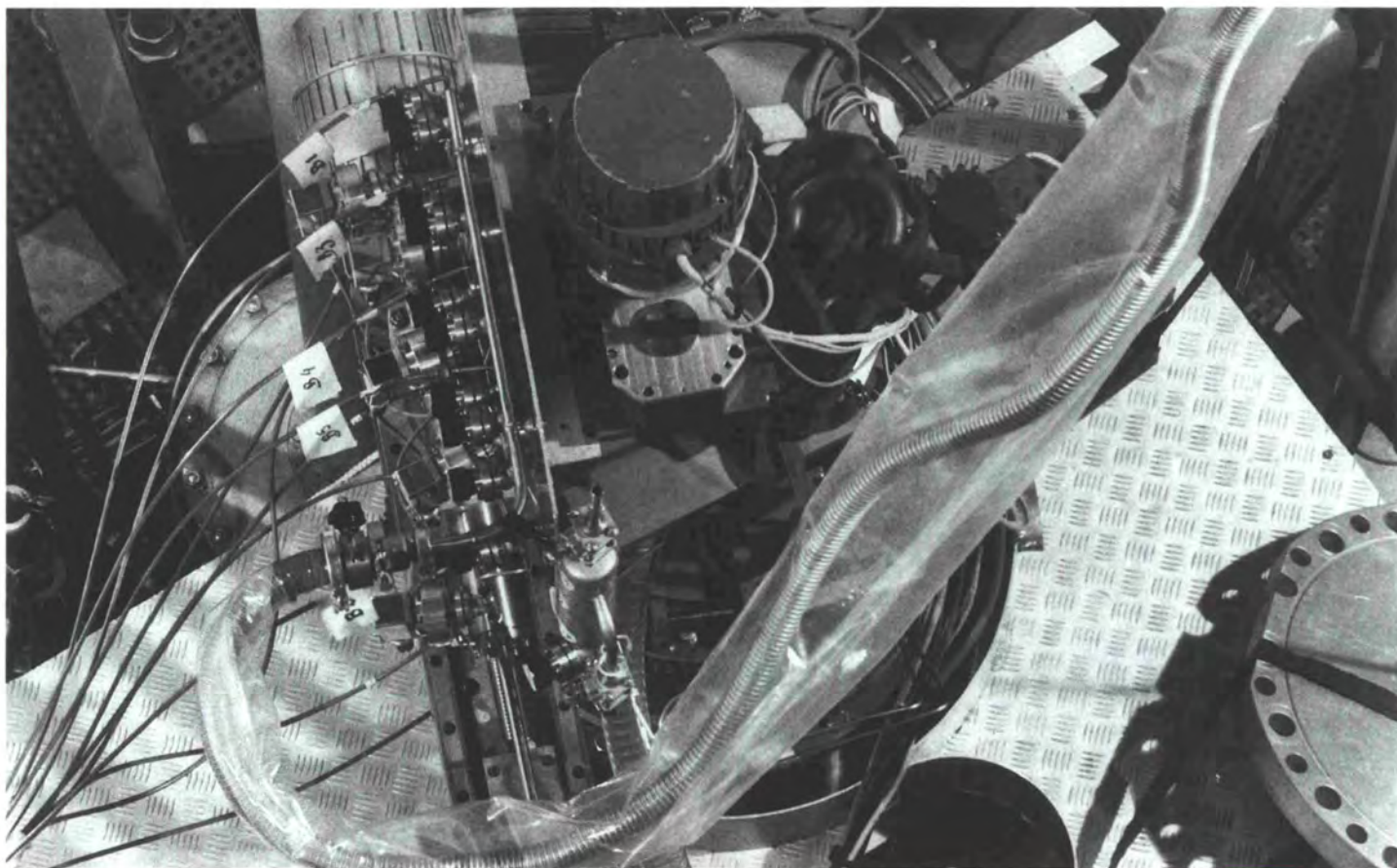
## CIRCUITS

The two processes of drying out the reflector tank, the first for changing to light water and the second to return to heavy water, were the two most delicate operations carried out by the circuits group. The maintenance of the purity of our stock of heavy water at 99.9% required these two operations to be very carefully carried out, which also necessitated considerable dismantling of pipe systems to disconnect the circuits changed to light water from the circuits retaining heavy water.

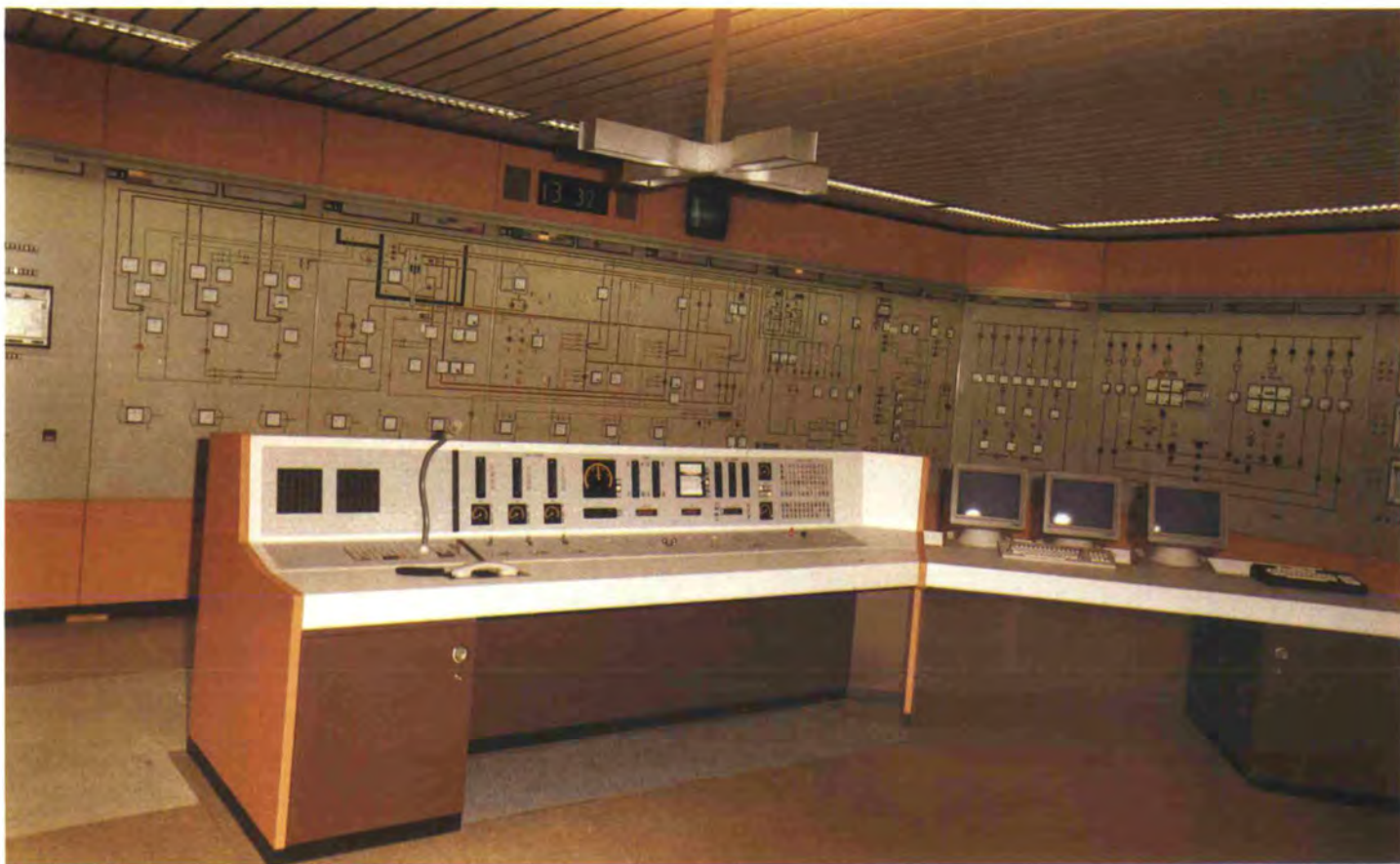
The shut-down was also used to carry out a verification of all the fluid circuits and to check the good condition of the main cooling exchangers of the reactor. The 'general compressed air' supply system was strengthened by the replacement of one of the three compressors by a new model capable by itself of providing all the normal supply to the installations.

## ELECTRICITY

The renovation of the alarm systems for the 'electrical distribution' and 'reactor building conditioning' installations took place in 1985, as did the installation of the new 'ARC BIS' no-break supply system for the experimental instruments. The lighting in the experimental hall of the reactor building was completely replaced and the high tension (15 KV) supply facilities extended.



*Control unit for the electro-erosion cutting device used for removing the remainder of the previous heavy water suction nozzle.*



*The rebuilt control panel of the HFR.*

## ELECTRONICS

The long shut-down also made it possible to replace all the thermo-dynamic measuring circuits, to verify all the neutron circuits ensuring the safety of the reactor, and to replace the Health Physics measuring equipment in the personnel protection circuit (tritium, dust, gas), while the operator's desk in the control room utilising the modular technique permitting adaptation to new components was completely rebuilt.

## HANDLING OF FUEL ELEMENTS

As a new neutron beam has been brought out in the upper level of the reactor, this necessitated the modification of the handling gantry for fuel element loading and unloading flasks. In addition to the systematic checking of the equipment, all the control relays for the handling circuits were replaced. This group also dealt with the cutting up in the hot cell of all the very active elements removed from the reactor block (beam tube thimbles) and the conditioning of the radioactive waste.

## OTHER WORK

### IMPROVEMENT OF THE AIR-TIGHTNESS OF THE REACTOR BUILDING

The linings in the lower part were partly replaced with a view to reducing the leak rate through the concrete shell.

At the level '204,25' the horizontal floor and the vertical wall have been completely repaired.

At the level '210,50' (reactor experimental hall) the horizontal part has also been completely repaired and the vertical concrete wall treated for a height of three metres.

A programme of work to be carried out to improve the air-tightness of the upper part of the reactor shell is in preparation and will be implemented during the year 1986.

## QUALITY ASSURANCE AND OTHER DOCUMENTS

A great deal of work on preparation of dossiers had to be carried out with the assistance of all the groups in the Reactor Department to up-date all the safety documents in order to obtain the authorisation of the Safety Authorities to restart the reactor on time.

## COLD SOURCES

### A. Vertical Cold Source

The new vertical cold source installed during the long shut-down was tested and started operation during the first cycle of reactor operation.

The aims of this project were as follows:

1. Increased neutron flux to the cold neutron guides H14 to H18.
2. Bringing of ultra-cold neutrons through the swimming pool into level D of the reactor hall.

The results obtained as regards these two aims are in accordance with or better than anticipated (improvement of 1.7 for guide H15, and of the order of 75 for the ultra-cold neutron flux in comparison with PN5).

However the increased cryogenic power consumed by the new vertical cold source results in the helium refrigerator operating at full power (two compressors in operation instead of one in the past). This resulted in an earlier request for installation of the third helium compressor, which had been planned in parallel with the commissioning of the future horizontal cold source, but which is now a priority requirement to maintain the operating reliability of the reactor. This compressor will be housed in an extension (still to be built) to the detritiation building.



Front view of the H1-H2 in-pile plug for the cold (above) and thermal guides. The new vertical Cold Source can be seen on the right.

### B. Horizontal Cold Source

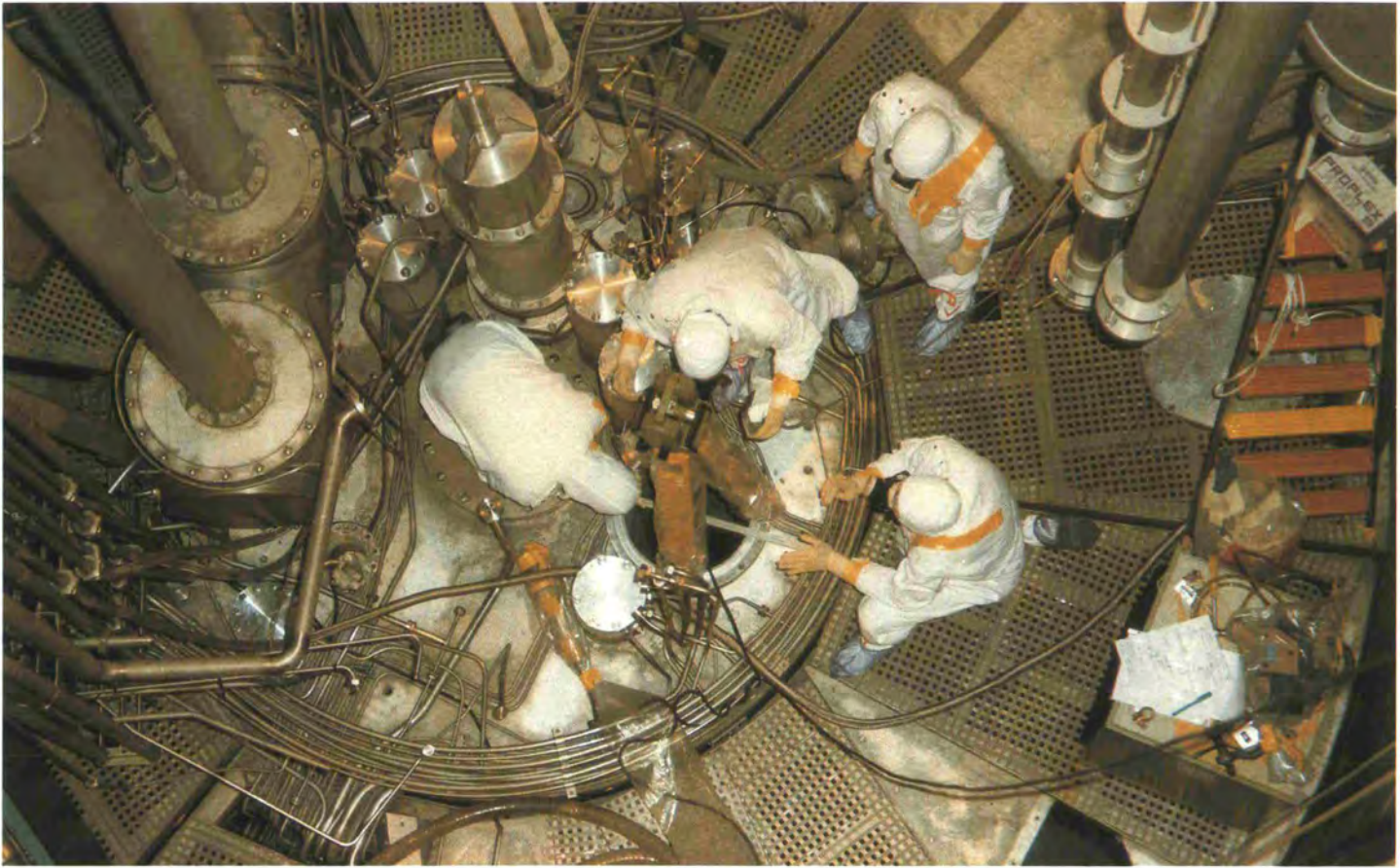
Work is continuing on the major orders already placed (cell, beam tube thimble, metal guide); other orders have been placed: plug, cold lines, condenser, beam shutter, safety valve.

The invitations to tender in progress are for the guides in the shutter and the deuterium buffer reservoir. Design studies are continuing for the facilities outside the reactor and for the controls and the pumping system to be built at ILL.

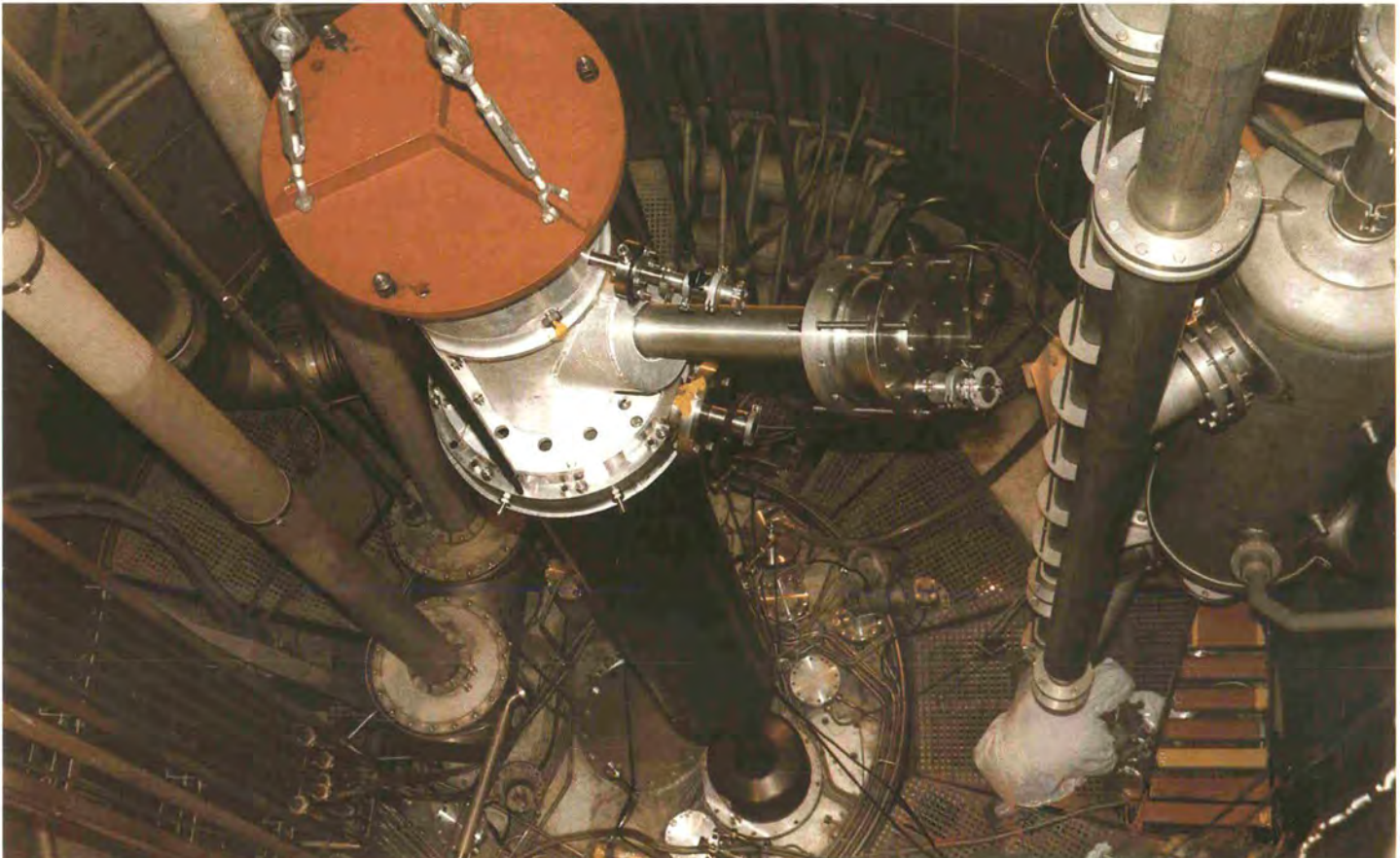
Present plans provide for installation in the reactor at the beginning of 1987.



Cutting device for the old H1-H2 plug-in unit.



*Alignment work for the new vertical Cold Source on top of the heavy water vessel.*



*The insertion of the new vertical Cold Source into the heavy water vessel.*

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**COMPUTING DEPARTMENT**

## **EINLEITUNG**

Der Reaktorstillstand bot eine wertvolle Gelegenheit, die ständige Routine der Abteilung zu unterbrechen. Einigen gab er die Zeit, seit langem nötige Instandhaltungs- und Konsolidierungsarbeiten in ihrem Verantwortungsbereich durchzuführen. Andere erweiterten ihre Berufserfahrung durch Besuche bei ähnlichen Laboratorien und Instituten, die unter normalen Umständen aufgrund des Arbeitsdrucks nicht möglich sind. Eine andere Gruppe gab oder organisierte Kurse über eine Vielzahl von Rechnerthemen für alle Mitarbeiter des ILL. Im Bereich der administrativen und wissenschaftlichen Informatik blieb die Arbeitsbelastung jedoch die gleiche, und die Belastung des Zentralrechners blieb bemerkenswert hoch.

Nach dem Wiederaufstart des Reaktors wurde der frühere Arbeitsrhythmus schnell wieder erreicht. Die Hauptaufgabe ist nun das Problem der rasch zunehmenden rechnergestützten Aktivitäten im gesamten Institut, die mit gleichbleibenden oder sogar reduzierten Mitteln bewältigt werden müssen.

## **INTRODUCTION**

The Reactor shutdown provided a valuable opportunity for interrupting the permanent routine of the Department. For some it offered the time to perform long needed maintenance or consolidation in their areas of responsibility. Others widened their professional experience by visits to associated laboratories and institutes, which, under normal circumstances, pressure of work does not permit. Another group gave or arranged lectures on a variety of computing topics for the benefit of all ILL staff. However in the areas of administrative and scientific computing the work continued much as usual, the load on the Central Computer remaining remarkably high.

Following the restart the former rhythm was quickly reestablished. The principal preoccupation is now the problem of handling the rapidly expanding computer-oriented activities, throughout the Institut, with constant or even reduced resources.

## **INTRODUCTION**

L'arrêt du Réacteur a fourni une occasion favorable d'interrompre le fonctionnement quotidien du Département. Pour certains ceci a permis d'effectuer enfin certains travaux essentiels de maintenance ou d'améliorer l'organisation des activités dans leur domaine de responsabilité. D'autres ont pu approfondir leur expérience professionnelle par des séjours dans des laboratoires ou instituts associés, ce que la charge de travail ne permet pas en temps normal. Un autre groupe a donné ou organisé des exposés sur divers sujets d'informatique à l'attention de tous les agents de l'Institut. Par contre, dans les domaines de l'informatique scientifique et de gestion, le travail a continué de façon à peu près normale et la charge de travail sur l'ordinateur central est restée remarquablement élevée.

Suite au redémarrage du Réacteur la cadence antérieure s'est rapidement rétablie. Le souci principal est désormais le traitement des activités informatiques, en pleine expansion dans tout l'Institut, et ceci avec des ressources constantes ou même réduites.

# **AUTOMATION AND DATA SERVICE (SAD)**

The Service is continuing to search for ways of improving its efficiency and performance. The three instrument software groups exist for reasons of organisational structure and reflect the different types of instrument in the 3 areas. But there is close contact between them and they work to agreed standards. To reinforce this policy it has been decided that all work on interfacing to VME, the new electronics bus, which will gradually replace CAMAC, will be centrally directed within the Service.

## **SOFTWARE A (CRYSTALLOGRAPHIC INSTRUMENTS)**

This Group is responsible for software for the instruments D1A, D1B, D2B, D3, D4, D5, D9, D10, D15, D16, D19B, D20, DB21, LI5, LI8, S18.

Having a large number of instruments located in the Reactor Hall, the Group was faced, during the Summer, with the task of reinstalling and testing all those computers that had been displaced during the shut-down. The Group is also, by chance, the one principally involved in the new instruments : D2B, D19B, D20, DB21.

On established instruments effort has been concentrated on the move towards standard operating systems (RSX on D1A and D3) and interfacing to new electronics (D1A, D4, D9, D10).

In collaboration with the scientists concerned, work continues to go into the treatment of single crystal data collected from large multidetectors, principally that of D19B. More sophisticated statistical techniques have been shown to be necessary if one wishes to extract the maximum of useful information.

An experimental ETHERNET network has been in operation throughout the year and has provided useful information on the performance to be expected from that protocol. It is intended to gradually extend this to become a full local area network, but costs will limit the rate at which this can be achieved. The Group also has responsibility for maintaining the DECNET based network, JUDAS, and for the DEC operating systems RSX, RT11, VMS.

## **SOFTWARE B (OTHER NEUTRON SCATTERING INSTRUMENTS)**

The group is responsible for the system and control software, and assists in data treatment for instruments in the THREE-AXIS and VERCORS groups (IN1, INFB, IN3, IN8, IN12, IN20, D7, D11, D17, IN4, IN5, IN6, IN10, IN11 and IN13).

The major activities of 1985 were in replacing computing systems on the small angle scattering spectrometer D11, and the triple axis spectrometer IN8. In both cases the systems were implemented with very effective contributions from the instrument scientists showing the advantages of the additional flexibility of the new computers.

In the case of D11 all software was rewritten or adapted to run on the new VAX 11/730 control computer, an exercise involving at various times most members of the group, and which served as a good introduction to the principal features of the VMS system. The low level CAMAC handler software reached a very stable state early in the year. New CAMAC libraries and the control program were progressively developed during the shutdown period, though this extensive work was only concluded satisfactorily when the whole instrument was available for tests after the reactor had restarted. Already the additional automation offered by the rebuild has been appreciated. Data transfer and local treatment have also been extensively rewritten for the new system. Planning for a similar computer system for D17 in 1986 is well advanced, and will re-use all this software.

The SOLAR 16/40 computer on IN8 was replaced by a PDP 11/73 based system running under the TSX monitor. Most software used here was based on that from IN20. The success of this collaboration with the physicists has led to plans to replace the remaining SOLAR computers on the three-axis spectrometers as soon as possible.

The remaining work performed by the Group concerned primarily the development of control routines for new facilities on IN5, IN12, IN13 and D17. The new chopper control and monitoring electronics for IN5 has required extensive programming for setting phases and speeds, and for following the temperatures of critical components. Part of this work is relevant to IN4. A novel mode of operation, scanning the monochromator angle rather than temperature has been successfully implemented on IN13.

## **SOFTWARE C (NUCLEAR PHYSICS AND SPECIAL INSTRUMENTS)**

On the Nuclear Physics instruments, the relatively limited user community, closely organised in large groups, makes the role of software support slightly different from that on the condensed matter instruments. The Group gives fullest support to the instruments PN2 (BILL), PN3 (GAMS 1,2,3), PN4, PN6 (OSTIS). PN1 and PN8 also receive support but are more under the responsibility of the scientists themselves.

A major effort is being made to adopt the appropriate standards, namely PDP 11/23+ and PDP 11/73 computers, TSX and RSX11M operating systems, DEC and Schlumberger CAMAC controllers. The data acquisition system on GAMS 1 has been completely reorganised, in collaboration with the instrument scientists. This new design is being implemented on PN4, and will also be applied to GAMS 2/3, when the latter instrument's new computer is installed in 1986. A new PDP 11/23+ was acquired for PN2, and installed with appropriate CAMAC controller towards the end of the year.

As many features of data treatment are common to all  $\beta$ - and  $\gamma$ -spectrometers their systems are linked to a local data treatment centre, where shared programs, graph plotter, graphics

terminals and magnetic tape facilities are available. This helps to offload the Central Computer, though access to the latter is provided, as elsewhere, for large-scale calculations and data archiving.

A number of instruments now use the "List Mode" technique in which event parameters are stored sequentially for analysis later. Some effort has gone into standardising the libraries of analysis programs, both in respect of instruments and computers.

Concerning the S instruments, the Group faces the same problem as other ILL support groups, namely that it wishes to help the scientists to achieve optimum performance from their instruments, but under ILL funding rules we have very limited resources to commit to such activities. The solution being adopted is to initiate discussions with the scientists responsible for building the instruments at the earliest possible time, principally to advise them as to the standards which are in operation at ILL. If they can agree to integrate computer systems which correspond to our norms (in respect of hardware, operating systems, peripheral drivers etc) then many problems can be avoided, and the Group may be able to provide useful assistance with relatively little perturbation to its principal activities.

The Group has acquired a Hewlett-Packard 9807 computer, a choice made on account of its IEEE interface, to see whether this could provide a low-cost solution to data acquisition on simple instruments. So far it has been used, in collaboration with the Electronics Service, for read-out and data transfer from multichannel analysers.

## MANAGEMENT INFORMATION SYSTEMS AND OFFICE AUTOMATION

The work of this Group can be roughly broken down into three areas

- the System MISSILL, an in-house design to handle services and functions peculiar to ILL, such as instrument scheduling and the visitor programme
- support for packages acquired under licence to handle normal administrative functions, such as salaries calculations.
- support for word-processing, both secretarial and scientific.

The major event in 1985 has been the installation of a VAX 11/750 to handle the principal word-processing service (work stations for about 12 secretaries, using the package OS11), the salaries package PACHA, which was successfully introduced at the beginning of the year, and a number of MISSILL-type facilities (such as for the medical and training sections) which overflowed from the PDP 11/34 on which the MISSILL system is installed. This load is close to saturating the VAX, and any future major projects will require further hardware investment.

A constant preoccupation is the rate of technical evolution in the field of word-processing. The system OS11 was chosen in 1982 as the best then available on DEC machines for both general and scientific text handling. Since that time a succession of products have appeared on the market specifically oriented towards scientific and mathematical text. One such system, MATHOR, which runs on the VICTOR personal computer, has been acquired for evaluation. A related point is that

rudimentary text-editing facilities are available on many of the Institut's computers, and one would like to be able to transfer the files so created for incorporation in text generated by secretaries. This problem is under active examination.

## COMPUTER HARDWARE MAINTENANCE

The Group spent the first half of the year in a systematic modernisation of the cabling from the instruments, the switchboards connecting these cables to the various computer and terminal networks, and of the rooms housing the service's own equipment. This was the first time since the start of the Institut that it had been possible to tackle this work, urgently needed because most modifications and additions had been made under pressure on an ad hoc basis. It is hoped to see the benefits of this in simplified access and maintenance. Subsequently, the Group had to reinstall all the instrument computers that had been displaced to permit the work in the Reactor Hall. This work, involving much cleaning and testing was hampered by the departure of a key technician, but by and large the time-table was respected.

There is considerable concern over the disequilibrium which is developing between the increasing amount of computing equipment in use at the Institut, and the limited resources (both money and manpower) available to maintain it. The problem is particularly evident in respect of terminals where, under the growing tendency of scientists to install terminals in their offices, combined with the rapid expansion of workstations for administrative and secretarial duties, the total number now exceeds 400. For computers themselves the problem is no less serious with the number installed seemingly growing without limit. Typically, an old computer which is no longer considered reliable enough to drive an instrument, rather than being scrapped, is kept in service for some less critical activity and remains on the maintenance load. The balance between in-house maintenance and external contracts is constantly under review, but it is not evident that our present standards of availability and reliability can be guaranteed in the future.

# CENTRAL COMPUTING SERVICE GRAPHICS

## OPERATION OF THE DEC-10

The DEC-10 operated with a high degree of reliability during the whole of 1985, i.e. with a rate of availability of 98 to 99%. The machine was available 24 hours a day with operator cover on Mondays from 5.30 to 21.30, and on Tuesday to Friday from 6.15 to 20.45. The loss of half an operator post in March 1985 resulted in the operator service on Saturdays being suspended. The scientists considered it desirable to restore this service after the Reactor start up to provide assistance to visitors on the ILL site at week-ends. A study to find a solution to this problem is in progress.

It may be noted that the degree of utilisation of the DEC-10 was relatively high (i.e. approximately 90% of the load during the two previous years) despite the absence of visitors and of a considerable number of scientists visiting external laboratories.

To resolve the problem of saturation encountered in 1983/84 we continued the investigation of an extension of the DEC-10 system. This operation proved more complex than expected, particularly following the announcement by DEC that they were stopping all development of new machines in the DEC-10/DEC-20 series. The negotiations with DEC took a considerable time and finally resulted in a proposal of a VAX 8600 with a simplified configuration, to be coupled with the DEC-10 for joint operation. This project was submitted to the Steering Committee and we received its approval at the end of the year.

## SYSTEM WORK AND COMMUNICATIONS

In the system software field, 1985 saw the updating of various software on the DEC-10 and PDP 11/24 UNIBUS peripheral server, i.e. the new monitors TOPS10-7.02 and RSX, the new DEC-10 DECNET and the new Fortran 77.

The installation of these items led us to modify a number of system programs, particularly the programs for information transfer between computers. Efforts have also been devoted to work on the VAX system for software and equipment not available on the DEC-10 and to a better integration of the Central Computer in the Transpac and equivalent networks.

We also examined the possibilities available with the EARN network, the main aim of which is to connect European universities and research centres in the context of message and file transfers between different types of machines. We are at present awaiting the hardware to permit connection to this system.

As regards the ILL network of terminals the installation of equipment to standardise input at the terminal switching device on the basis of two standard speeds of 2400 and 9600 bauds should be noted.

At the beginning of the year the ILL received a vectorial type graphic system, the Evans & Sutherland PS330, purchased and used jointly with EMBL. This is connected to a VAX 11/750, as all the software obtained from various universities (Rice and Cambridge) is written for the VAX. Apart from the Fortran and Pascal libraries, intended for developments of this system, we have installed FRODO and HYDRA software for displaying molecular structures for applications in crystallography and biology. We have also developed two display programs for multidetector spectra with possibility of local interaction with the spectra.

In addition to the PS330, the Service has purchased a Raster-type colour graphics terminal aimed at applications requiring some processing of images such as detector matrix spectra.

A study has also been carried out of the hard-copy market to identify equipment capable of receiving images from these screens with acceptable definition and colour range.

As regards the basic graphics software accessible to the scientists, a standard has just been adopted by all the manufacturers of graphics hardware - the GKS library. We have acquired this for the VAX and DEC-10, and it will be the only interface for all types of graphics hardware at ILL.

## MATHEMATICS

This year we have installed on a VAX 11/750 the Harwell library subroutines generally used on the DEC-10, and Fortran program development tools (TOOLPACK) ; these permit in particular analysis of language and program analysis during operation.

There is currently a demand for the purchase of a library more complete than the Harwell one. Following a study carried out to compare NAG and IMSL, the former was chosen.

## COURSES, SEMINARS, INFORMATION, DISSEMINATION

In connection with the Reactor shutdown, the Service organised a number of courses and seminars on various subjects :

- Fortran 77, DEC-10 and VAX
- Mathematical software available at ILL, methods of program optimisation
- Introduction to Pascal
- Seminar on statistics
- Graphics software (on PS330, NAG graphics)
- Information on the TOPS10 operating system.

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**ADMINISTRATION DEPARTMENT**

## EINLEITUNG

Man könnte annehmen, dass das Jahr 1985 für die Verwaltung ein ruhiges Jahr war, da der Reaktor wegen der Erneuerungsarbeiten bis zum September nicht in Betrieb war. Dessen ungeachtet hatte die Verwaltung jedoch eher ein sehr betrieb-sames, ereignisreiches Jahr.

Nach dem Ablauf der Amtszeit des bisherigen Direktors, Dr. Fender und des deutschen stellvertretenden Direktors, Prof. Ruppertsberg, wurden von der neuen Direktion die Planungen für den Haushalt 1986 fortgeführt. Sie bestätigten trotz aller Bereitschaft zur Sparsamkeit, dass es unmöglich sein würde, die Ausgaben des Normalbudgets 1986 gegenüber 1985 auf real konstantem Niveau zu halten.

Die hauptsächlichsten Gründe für die Ausgabenerhöhung sind :- Notwendigkeit, die Folgekosten des 1985 zu Ende gegange-nen Modernisierungsprogramms ab 1986 im Normalbudget zu decken

- Rückkehr zu einem vollen Betrieb des Reaktors und der Ex-perimentieranlagen

- Erweiterung der Kapazität der zentralen Rechenanlage

- Entwicklung von Instrumenten zum Anschluss an die im Bau befindliche neue Kalte Quelle.

Die Gesellschafter setzten eine aus 6 Wissenschaftlern beste-hende Gutachter-Kommission ein, um den Ausgabenbedarf des ILL zu durchleuchten. Die Kommission kam ebenfalls zu der Ansicht, dass auch bei Ausschöpfung aller Sparvorschläge das Budget des ILL 1986 und 1987 erhöht werden müsste.

Die Gesellschafter haben daraufhin beschlossen, ihre Zuschüs-se zum Normalbudget 1986 um rund 15 Mio F auf 245 Mio F zu erhöhen. (Geldwert 1985, ohne Steuern.)

## INTRODUCTION

It might be assumed that 1985 was a quiet year for the Ad-ministration, as the Reactor did not operate until September because of the renewal work. In fact the Administration had rather a busy, eventful year.

On expiry of the period of office of the previous Director, Dr. Fender, and the German Assistant Director, Professor Rup-pertsberg, the new Directors continued the planning for the 1986 budget. They found that despite the will to economize, it would be impossible to maintain the expenditure under the 1986 normal budget at a real constant level in comparison with 1985.

The main reasons for the increased expenditure are :

- The need to cover the costs resulting from the Modernisa-tion Programme, completed in 1985, in the Normal Budget from 1986.

- Return to full operation of the reactor and the experimental facilities.

- Enhancement of the Central Computer capacity.

- Development of instruments for connection to the new Cold Source currently under construction.

The Associates set up a commission consisting of 6 scientists to examine the financial needs of the ILL. The commission also came to the conclusion that even if all the proposed economies were implemented the ILL budget must be increased in 1986 and 1987.

The Associates then decided to increase their grants for the 1986 Normal Budget by approximately 15 MF to 245 MF (at 1985 prices, without taxes).

## INTRODUCTION

On pourrait penser que 1985, l'année où le Réacteur a été arrêté jusqu'au mois de septembre pour mener à bien les travaux de rénovation, a été une année tranquille pour l'Administration. Bien au contraire, et malgré cet arrêt, 1985 a été une année bien remplie.

A l'expiration des mandats du Directeur alors en fonction, M. Fender et du Directeur-Adjoint allemand, M. Ruppertsberg, la nouvelle Direction de l'Institut a poursuivi les travaux d'éta-blisement du Budget 1986. Ceux-ci ont confirmé qu'en dépit d'une volonté d'économies, il serait impossible de maintenir le volume de dépenses du Budget Normal 1986 à un niveau constant par rapport à 1985.

Cette augmentation s'explique essentiellement pour les raisons suivantes :

- A partir de 1986, le Budget Normal devra couvrir les frais induits par le Programme de Modernisation;

- Le Réacteur et les dispositifs expérimentaux ont repris leur fonctionnement normal;

- La capacité du Calculateur Central devra être augmentée;

- Les instruments destinées à la nouvelle Source Froide, actuel-lement en cours de construction, devront être mis au point.

Les Associés ont créé une commission de 6 experts chargée d'examiner les besoins financiers de l'ILL. Cette commission est également parvenue à la conclusion que, malgré toutes les propositions d'économies, le Budget de l'ILL devrait être aug-menté en 1986 et 1987.

C'est pourquoi, les Associés ont accepté d'augmenter leurs contributions au Budget Normal 1986 d'environ 15 MF en le fixant à 245 Millions de F (valeur 1985, hors taxes).

# FINANCE

## IMPLEMENTATION OF THE 1985 BUDGET

### Normal Budget

The 1985 Normal Budget was authorized with the following totals:

expenditure	237.2 MF )	
income	7.8 MF )	(excluding taxes)
Associates' grants	229.4 MF )	

The reduction in the number of cycles to 3 (instead of 6 in a normal year) permitted savings which were used mainly for the continued reactor renewal programme and the equipment of a second neutron guide hall in which the instruments on the new Horizontal Cold Source will be installed in 1987.

In comparison with the previous year the operation expenditure increased from 193.2 MF to 204 MF (+ 5.6 %) which in real terms corresponds to a growth of 1.5 %. If staff costs (2.9% higher) and the expenditure on fuel elements (which increased in preparation for the return to normal reactor operation) are excluded from this comparison, the 1985 operation budget (49.3 MF) is 7 % less than the figure for 1984 (52.7 MF). Investments increased by 3.2 % (cf. table).

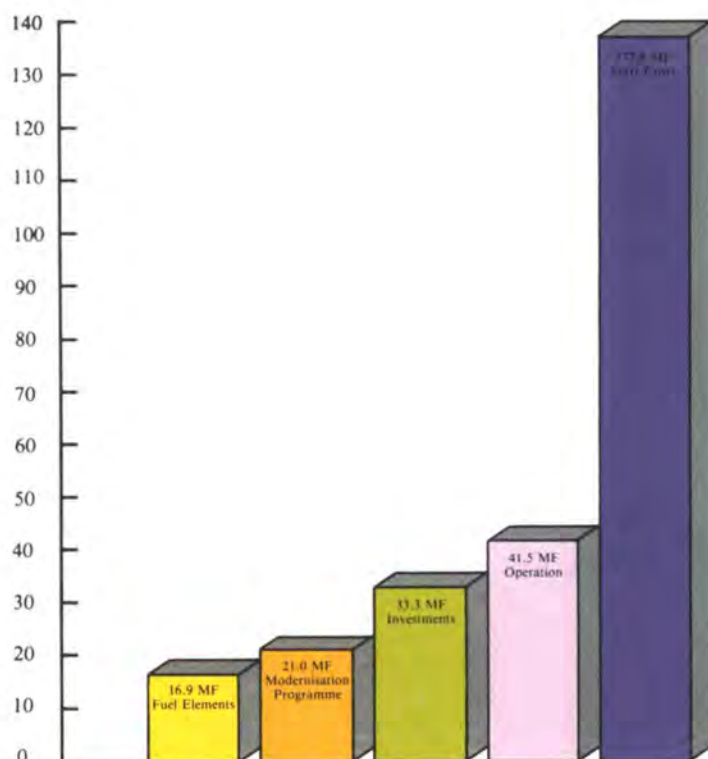


Figure 60 : Associates' Contributions 1985 : 250,5 MF

### Comparison in expenditure in 1984 and 1985 (normal budget)

	1984		1985	
	(1 000 F)	% of total	(1 000 F)	% of total
a) Operation				
Consumable materials	15 973	7.1	21 809	9.2
Fuel elements	11 828	5.2	16 925	7.2
Staff costs	128 696	57.1	137 785	58.3
Taxes	1 340	0.6	1 260	0.5
Long term service & supply contracts	17 544	7.8	8 912	3.8
Other work, supplies & services	9 756	4.3	11 091	4.7
Transport, removal & travel expenses	2 044	0.9	1 810	0.8
Miscellaneous administr. costs	6 018	2.7	4 399	1.9
<b>Total operation</b>	<b>193 199</b>	<b>85.7</b>	<b>203 991</b>	<b>86.0</b>
b) Investments				
Buildings	2 773	1.2	976	0.4
Equipment	10 909	4.9	8 891	3.7
Experimental instruments	12 933	5.7	12 713	5.4
Other investments	5 608	2.5	10 675	4.5
<b>Total investments</b>	<b>32 223</b>	<b>14.3</b>	<b>33 255</b>	<b>14.0</b>
<b>Total expenditure</b>	<b>225 422</b>	<b>100.0</b>	<b>237 246</b>	<b>100.0</b>
c) Income				
ILL's own income	7 907	3.5	7 795	3.3
Grants from Associates	217 515	96.5	229 451	96.7
<b>Total income</b>	<b>225 422</b>	<b>100.0</b>	<b>237 246</b>	<b>100.0</b>

### Modernisation Programme

The complete Modernisation Programme planned to run from 1979 to 1985 was evaluated at 104.2 MF (1979 prices). At the end of 1985 projects to the value of 95.4 MF (at 1979 prices), 133 MF (actual expenditure) had been completed. Only the design studies and construction of the new Horizontal Cold Source will be continued and probably completed in 1987.

The figure for 1985 for the Modernisation Programme amounts to 21.02 MF, plus 5.3 MF not utilized in 1984 which was brought forward. Of the total thus available of 26.3 MF, 11 MF was used in 1985. The balance, entirely reserved for the Horizontal Cold Source project, will be carried forward to 1986.

The breakdown by section of the expenditure is as follows :

### Budget for the Modernisation Programme

(excluding taxes)

	1985 (1 000 F)	% of total
a) Operation		
Consumable materials & small equipment	540	4.8
Staff costs	4 500	39.9
Long-term service and supply contracts	130	1.2
Other work, supplies & services from third parties	1 940	17.2
Transport, removal & travel expenses	160	1.4
Miscellaneous administrative costs	190	1.7
<b>Total operation expenditure</b>	<b>7 460</b>	<b>66.2</b>
ILL's own income	- 1 820	- 16.1
<b>Total operation</b>	<b>5 640</b>	<b>50.1</b>
b) Investments		
Experimental instruments	5 625	49.9
<b>Total expenditure</b>	<b>11 265</b>	<b>100.00</b>
Carry forward to 1986	15 103	
<b>Total</b>	<b>26 368</b>	

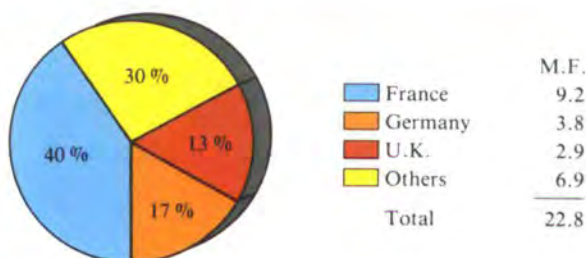


Figure 61: Distribution of ILL's purchases.

### PURCHASING

Considerable progress has been made in ordering the components for the New Horizontal Cold Source. The Zircalloy beam tube liner is being manufactured in France by CERCA and the nickel neutron guide block will be constructed in Germany by NTG. The alignment plug of the Cold Source will be made in England by Cumbria Engineering. Smaller value orders have also been placed for the safety valve, the cryogenic transfer lines and the connecting bellows to the shutter. The exchanger-condenser will be constructed in France by Air Liquide. Invitations to tender for the compressor have already been sent out.

The Institut purchased 25 kg of Uranium 235 from the KfK for delivery to CERCA and NUKEM for the manufacture of fuel elements to be supplied in 1986 and 1987.

To cover our requirements for heavy water, the ILL has purchased 1 000 litres of heavy water with a tritium content of 14 Curies from the CEA Brennilis. After processing in the ILL detritiation plant, it will be used in the High Flux Reactor.

In order to cope with the ever-increasing data from our experiments it has become necessary to extend the capacity of the central computer (a DEC 1091), and therefore a VAX 8600 has been ordered. It will be installed in March 1986. A VAX minicomputer was purchased for D11 and after successful use on this instrument, a similar VAX system has been ordered for D17. As both instruments have the same group of users and similar functions software developed on D11 can also be used on D17.

PLESSEY continues to be the ILL's most important British supplier with orders for minicomputers, terminals and other peripherals reaching a total of 1.6 MF in 1985.

The demand for ILL precision temperature controllers continues, and an order was placed with DUHAMEL, for 12 controllers. A further 5 controllers have been ordered for ILL's clients and new demands are expected next year both for ILL instruments and for users in other research centres.

As in previous years, the ILL ensured that firms in the three member states were invited to tender for all major purchases. ILL engineers therefore also visited British and German companies to discuss technical problems, and to establish contacts with new suppliers. In this respect the KfK and RAL also assisted by suggesting suitable suppliers in their respective countries.

The distribution of ILL's 'free' purchases\*) in 1985 is shown in the diagram.

	MF	%
France	9.2	40
UK	2.9	13
Germany	3.8	17
Others	6.9	30
	<b>22.8</b>	<b>100</b>

\*) 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.

# PERSONNEL

## STAFF

The year 1985 was characterized by the first specific measures due to the completion of the Modernisation Programme.

The staff authorized was reduced from 508.5 posts in 1984 to 506 in 1985. The staff occupying posts went down from 497.5 on 31/12/84 to 494.5 on 31/12/85. The authorized and actual staff will continue to diminish until the 17.5 posts granted under the Modernisation Programme have been completely absorbed. The plan prepared by the ILL Management and the expected retirements should permit this aim to be achieved on schedule.

There were slightly more staff movements in 1985 than in 1984 (7.5 % as against 7 %). This was mainly due to the considerable number of thesis students leaving during the year. Almost all the recruitments were for the scientific sector (physicists and thesis students). As regards scientists on limited term contracts, the situation was clarified by the publication of decree 85-389 of 27/03/85 permitting the signature of limited term contracts in scientific research carried out under an international agreement.

There was no significant change in comparison with 1984 in the breakdown of staff by sex (17 % women, 83 % men), and nationality (64 % French, 15.3 % German, 15.6 % British, 5.1 % others). The average age of the staff was 39.8 years (39 in 1984).

## Industrial Relations and Salaries

The elections of the Works Committee (Comité d'Entreprise) and Staff Representatives (Délégués du Personnel) were held early in the year. The results for the "Cadres" showed relative

stability with the CGC maintaining its majority, and for the "Non-Cadres" an increase in the representation of FO, which thus becomes the largest union for this category of staff.

The agreements resulting from the laws on industrial relations passed in 1982 and 1983 were generally well accepted. However 2 years after its introduction the employees' right of expression has not had the success expected among the staff.

The law on the obligation to negotiate salaries made official the existing periodic meetings between the Administration and the unions, without really changing the situation in that the ILL is required by its statutes to follow salary agreements signed at the CEA.

The corresponding salary agreement for 1985 resulted in a general increase in salary levels of 4.5 %.

Negotiations on other important points, such as the rates of advancement and the situation of shift staff were also initiated. This was in fact a matter of adapting CEA provisions for the ILL.

## Work during the reactor shutdown

The figure for guest scientists visiting the ILL does not appear useful this year in view of the reactor shutdown for more than 9 months.

During this shutdown, other opportunities arose for the ILL staff. A certain number of scientists and to a lesser extent technicians were able to use and enhance their knowledge on detachment in the laboratories of associates or universities. Another group of staff, particularly in the Reactor Department, was in great demand for work associated with the shutdown. Appropriate solutions to deal with particular working arrangements (hours, conditions, payment) were implemented either with the agreement of the unions or after consultation of the Works Committee, in accordance with existing regulations.

Finally the new computerized salary and personnel management system, introduced on 1 January 1985, has proved successful after one year's operation, and sincere thanks are due to all staff who assisted with this work.

## Staff Movements (on posts) in 1985

CATEGORIES	POSITION ON 31/12/84	RECRUITMENT	DEPARTURES	DIFFERENCE	POSITION ON 31/12/85	CHANGE % COL. 4 COMPARED WITH COL.2
	(2)	(3)	(4)	(5)	(6)	
SCIENTISTS	73	15	18	- 3	70	24.7
ENGINEERS	56	-	-	-	56	-
OTHER 'CADRES'	11.5	2	2.5	0.5	11	21.7
THESIS STUDENTS	23	14	12	+ 2	25	52.2
TECHNICIANS	174	-	3	- 3	171	1.7
OTHERS	160	3.5	2	+ 1.5	161	1.3
<b>TOTAL</b>	<b>497.5</b>	<b>34.5</b>	<b>37.5</b>	<b>- 3</b>	<b>494.5</b>	<b>7.5</b>

# WELFARE + GENERAL SERVICES

## HOUSING

The "Maison des Universités" project at Grenoble was finally completed in 1985 : it is situated in the Hoche Centre in Grenoble, near the town centre. It comprises some 50 flats intended for the University of Grenoble I, ILL, IRAM, INPG (Institut National Polytechnique de Grenoble) and the Ecole Supérieure de Commerce.

The ILL has reserved 5 flats (by paying a contribution) which we have furnished and equipped for renting to young thesis students. The ILL now has 16 furnished flats intended for guest scientists and new staff.

## EDUCATION

The international "Houille Blanche" primary school has 126 children, about half of whom are of German or English mother tongue. The "Eaux Claires" secondary school also has some 60 children in its "international sections".

Studies are in progress for the establishment at Grenoble of a school specifically established to provide an international secondary education.

There are close contacts between the ILL Management and the local and national authorities concerned; vigilance must be maintained, particularly in the light of the establishment of the European Synchrotron Radiation Facility.

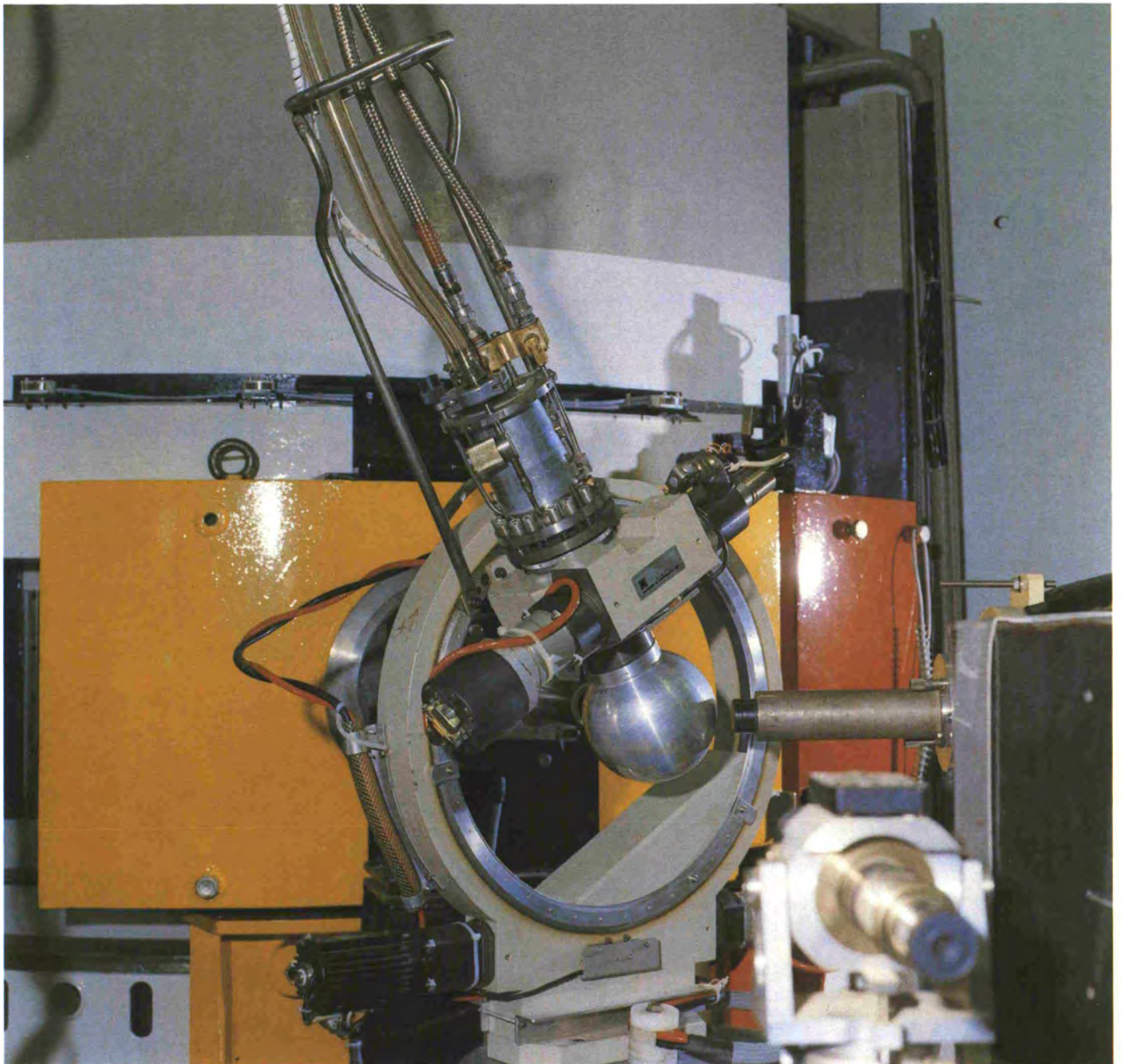
## STAFF TRAINING

There were almost 12 000 hours of training in 1985, corresponding to 217 courses, which is an average course duration of 52 hours. The courses in 1985 were mainly on computing, micro-computers, programmable control devices, statistical methods, etc.

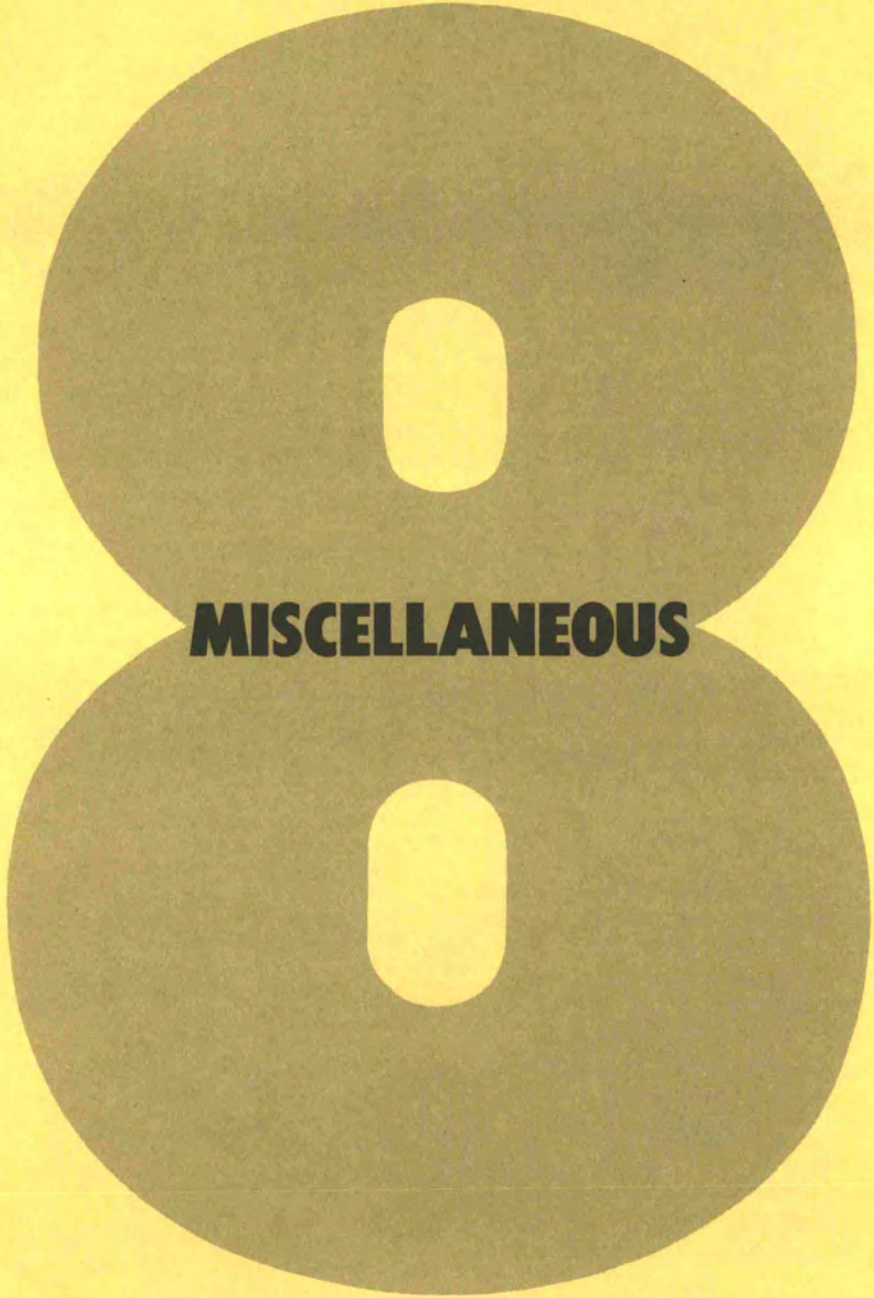
As last year, there has been an increasing demand by staff for training in their professional field.

## MEDICAL SERVICE

Since the beginning of 1985, the secretariat of the medical service has been computerized. Previously about 6 000 appointment forms for medical examinations were completed by hand each year, plus reminders. Computerization has meant that numerous files, schedules etc. no longer need to be maintained.



*The recently reconstructed four-circle diffractometer D9 located on the hot beam tube H3.  
The photograph shows the new two-stage cryorefrigerator mounted on the offset Eulerian cradle allowing for routine high-resolution structural work down to 15 K.*



**MISCELLANEOUS**

## LIST OF SEMINARS

### COLLEGE 2: THEORY

- "Multielectron dimples in liquid and solid Helium".  
V. SHINKIN, Institute of Solid State Physics, Moscow.
- "Percolation and superconductivity".  
G. DEUTCHER, Université de Tel-Aviv.
- "Superconductivity and charge ordering in the negative U Hubbard model".  
S. ROBASZKIEWICZ, A. Minkiewicz University, Poznan (Poland).
- "The Chapman - Enskog method for the relativistic Boltzmann equation".  
N.G. VAN KAMPEN, Aachen.
- "Exposants critiques loin de la transition dans quelques systèmes magnétiques".  
J. SOULETIE, CRTBT, CNRS, Grenoble.
- "Transition d'Anderson sur le réseau de Bethe: résultats exacts".  
H. KUNZ, Ecole Polytechnique de Lausanne.
- "Structure des amorphes et espaces gaufrés".  
J.P. GASPARD, Université de Liège.
- "Dynamics of diluted Ising systems".  
C.K. HARRIS, Université d'Edimbourg.
- "Generalized many channel Landauer formula".  
Y. IMRY, Université de Tel-Aviv.
- "Phonon linewidths in local level systems".  
J.G. HUGGINS, Max-Planck Institute, Stuttgart.
- "Les théories super-symétriques des particules".  
P. FAYET, Ecole Normale Supérieure, Paris.
- "Finite size scaling and variational calculations for quantum spin systems".  
J. SOLYOM, Central Research Institute, Budapest et Université de Lausanne.
- "Characterization of dynamical instabilities in simple chaotic systems".  
H. SCHUSTER, Université de Francfort.
- "Transition d'Anderson dans les systèmes désordonnés et les fractals : une approche à la Borland".  
B. SOUILLARD, Ecole Polytechnique, Palaiseau.
- "Mésons, baryons et hadrons exotiques dans le modèle des quarks".  
J.M. RICHARD, I.L.L.
- "Diffusive transport in colloidal suspensions".  
R.B. JONES, Queen Mary College, Université de Londres and I.L.L.
- "Lippmann-Schwinger theory of half-collision cross sections".  
M. GIRARDEAU, Max-Planck Institut für Strahlenchemie, Mülheim.
- "A random walk model for diffusion in supercooled liquids and glasses".  
W. SCHIRMACHER, I.L.L.
- "Etats de Stark-Wannier dans les cristaux et les solides désordonnés".  
F. BENTOSELA, Centre de Physique Théorique, CNRS, Marseille.
- "Dendritic solidification patterns".  
J. LANGER, Institute for Theoretical Physics, Santa Barbara.
- "Magnétisme et supraconductivité dans les systèmes de fermions lourds".  
S. DONIACH, Stanford University and CEN Saclay.
- "Roughening transition and grain boundaries".  
C. ROTTMAN, Ohio State University.
- "Diffusion anormale et bruit 1/f dans les systèmes chaotiques".  
T. GEISEL, Universität Regensburg.
- "Heavy Fermi liquid: an 'almost broken symmetry'".  
P. COLEMAN, Santa Barbara and Cambridge.
- "Etude théorique de la transition de phase dans les fluides Coulombiens : cas des mélanges métal-sel fondus".  
G. CHABRIER, Département de Physique des Matériaux, Université de Lyon.
- "Localisation du gaz d'électrons classiques dans un champ périodique : un exemple de transition de Kosterlitz-Thouless".  
J.P. HANSEN, Laboratoire de Physique Théorique des Liquides, Université Pierre et Marie Curie, Paris.
- "Spin glasses with random anisotropy".  
K. FISCHER, Jülich.
- "Phase transition in spin systems : quantum effects and random fields".  
R. MICNAS, Université de Poznan.
- " $\epsilon$ -expansion for finite size scaling with free boundaries".  
E. EISENRIEGLER, Jülich.
- "Diffusion in lattice gas models".  
H. VAN BEIJEREN, Institut für Theoretische Physik, Aachen.
- "Déformations des ondes de densité de charge".  
M. RENARD, CRTBT, CNRS.
- "Electron correlations in magnetic states of transition metals".  
A. OLES, Max-Planck Institut, Stuttgart.

"The Wigner distribution and linear response formula for the Hall coefficient".

J. MORGAN, Physics Department, Leeds University.

"Magnetic excitations in itinerant electron materials".

H. MOOK, Oak Ridge National Laboratory.

"Theory of normal heavy fermion systems".

B. BRANDOW, Los Alamos National laboratory, New Mexico.

"Supraconductivité triplet et magnétisme itinérant".

H. CAPELLMANN, I.L.L.

"Théories du champ sur réseau".

J.M. DROUPPE, Service de Physique Théorique, C.E.N. Saclay.

"Is liquid  $^3\text{He}$  nearly metamagnetic?"

K. BEDELL, University of Leiden.

"Thermal exchange between atomic hydrogen gas and superfluid Helium surfaces".

V. GOLDMAN, University of Amsterdam.

"Similarity operators: a new tool in crystallography".

E. BERTAUT, Laboratoire de Cristallographie, CNRS, Grenoble.

### COLLEGE 3: FUNDAMENTAL AND NUCLEAR PHYSICS

"Search for a neutron electric dipole moment".

P. MIRANDA, I.L.L.

"Extension of supersymmetries in the interacting boson model".

P. VAN ISACKER, Gent.

"The level structure of light actinides:  $^{215}\text{Fr}$  and  $^{219}\text{Ac}$ ".

J. CIZEWSKI, Yale.

"Phase space formulation of quantum mechanics for time dependent processes".

H. KOHL, University of Frankfurt.

"Experiments on time reversal invariance".

K. SCHRECKENBACH, I.L.L.

"Synchrotron radiation activities at the NPI of Novosibirsk: Microlithography, holography, microscopy with X-rays. Experimental techniques (wigglers, detectors)".

E. GLUSKIN, NPI Novosibirsk.

"Semiclassical model of multi-step direct and compound nuclear reactions".

ZHUO YIZHONG, Institute of Atomic Energy Beijing (China).

"Small drops of saturating matter".

J. TREINER, Orsay.

"Experiments with Compton backscattered Laser photons at the electron storage ring VEP 4 in Novosibirsk".

V. NEDOREZOV, Gif-sur-Yvette.

"Variation of even-odd charge effects with excitation energy in  $\nu(A)$  and mass yield for  $^{252}\text{Cf}(sf)$ ".

R.L. WALSH, CEN Bruyères-le-Châtel.

"Study of the  $^{233}\text{U}(n,f)$  process on Lohengrin".

U. QUADE, TU München.

"Low temperature scanning electron microscopy: a new way to see structures in solids".

R.P. HUBENER, University of Tübingen.

"Atomic physics below  $10^{-22}$  eV: electric dipole moments and tests of Lorentz invariance".

B. HECKEL, University of Washington.

"Cold fragmentation in fission".

F. GÖNNENWEIN, University of Tübingen.

"Collective excitations - the work of college 4".

B. STIRLING, I.L.L.

"Neutrino mass measurements".

T. BOWLES, Los Alamos National Laboratory.

"The new vertical source of cold and ultracold neutrons at the I.L.L.".

A. STEYERL, TU München.

"Identification of Nilsson states in  $A \approx 100$  nuclei by investigation of gross beta-decay properties".

K.L. KRATZ, University of Mainz.

"Search for parity violation in resonance scattering of gamma rays".

A. BEDA, ITEP Moscow.

"Radiative capture reaction induced by fast neutrons".

S. JOLY, CEA Bruyères-le-Châtel.

"Weak interaction physics at Argonne and neutrino oscillations at LAMPF".

S. FREDMAN, Argonne National Laboratory.

"New collective modes in proton-neutron systems".

K. HEYDE, University of Gent.

"A new unified approach to nuclear structure: the  $N_p N_n$  scheme".

R.F. CASTEN, Brookhaven National Laboratory.

"High resolution X-ray and particle detection with superconductive tunnel junctions".

V. ZACEK, CERN, Genève.

"Hydrogen tunneling in  $\text{Nb}(\text{OH})_x$ ".

A. MAGERL, I.L.L.

"P-odd effects in slow neutron fission".  
G.A. PETROV, NPI Leningrad.

#### COLLEGE 4: STRUCTURAL AND MAGNETIC EXCITATIONS

"Anomalous transport properties near phase transitions in charge-density-wave systems".  
M. NUNEZ-REGUEIRO, B.T.-CENG.

"Diffuse X-ray scattering from interfaces in crystals".  
S.R. ANDREWS, Gen. Electric Research Laboratory, Wembley.

"The liquid-solid interface".  
H.Z. CUMMINS, City College, New York and Université Paris VI.

"Random alloys : from Schrödinger's equation to the rolling mill - photoemission studies of their electronic band structure and magnetic properties".  
R.G. JORDAN, University of Birmingham.

"Critical reflection of polarized neutrons".  
J.A.C. BLAND, I.L.L.

"Flux quantization in superconducting and normal metal networks".  
B. PANNETIER, CRTBT, CNRS.

"Neutron scattering studies of a dilute anti-ferromagnet  $Cd_{1-x}Mn_xTe$ ".  
U. STEIGENBERGER, I.L.L.

"Heavy fermion superconductors".  
J.J.M. FRANSE, Université d'Amsterdam and Laboratoire Louis Néel.

"Calorimetric investigations of series of field-induced spin-density-wave states in a quasi-1D metal".  
P. GAROCHE, Laboratoire de Physique des Solides, Orsay.

"Static and dynamic properties of the soliton lattice in incommensurate crystals".  
H.G. UNRUH, Universität Saarbrücken.

"The onset of ferromagnetism in PdNi".  
E. LINDLEY, I.L.L.

"I R I S - A high resolution quasi elastic and inelastic spectrometer on the spallation neutron source".  
R. WARD, Rutherford Laboratory.

"Quasi-crystalline phases : example of the icosahedral structure in rapidly solidified Al-Mn alloys".  
D. GRATIAS, CECM, Vitry.

"Nucleation, growth and scaling in a pressure-induced first-order phase transformation: RbI".  
J.D. AXE, Brookhaven National Laboratory.

"Phonon anomalies in tetrahedral framework structures".  
G. ECKOLD, KFA Jülich.

"Critical and multicritical wetting phenomena in systems with long-range forces".  
W. SAAM, Ohio State University and ENS-Paris.

"Lattice dynamics of bromine intercalated in graphite".  
C. SIMON, G.P.S., Paris VII.

"Localization in incommensurate and random systems".  
R. PRANGE, University of Maryland.

"X-ray absorption studies on mixed-valent materials using synchrotron radiation".  
G. KAINDL, F.U. Berlin.

" $\mu$ eV - resolution on a 3-axis spectrometer- (experiments in Brookhaven)".  
A. HEIDEMANN, I.L.L.

"The pulsed neutron source constant-Q spectrometer".  
R.A. ROBINSON, LANL, Los Alamos.

"Spin waves in  $K_2Co_xFe_{1-x}F_4$  : computer simulations and experiments".  
M. HAGEN, Oak Ridge National Laboratory.

"Spectroscopic generation and dynamics of nonequilibrium phonon pulses".  
K.F. RENK, University Regensburg.

"Recent neutron experiments at Hahn-Meitner-Institut and Brookhaven".  
G. SHIRANE, Brookhaven National Laboratory.

"Dielectric spectroscopy in solids :  
1. Soft-modes in ferroelectrics  
2. Superionic conductors".  
J. PETZELT, Prague.

"Electronic properties of metal hydrides : from metal-semiconductor transition to heavy fermion behaviour".  
L. SCHLAPBACH, ETH-Zürich.

"Neutron scattering studies of heavy fermion systems".  
W.J.L. BUYERS, AECL-Chalk River.

"Spinodal decomposition in  $NbH_x$  : neutron radiography and dilatometric studies".  
G. STEYRER, I.L.L.

"Phonon spectroscopy with superconducting junctions".  
H. KINDER, TU-Garching.

"Fermi surface studies and quantum fluctuation in praseodymium".  
M. WULFF, JRTE-Karlsruhe.

"Solving surface structure problems with X-rays".  
R. FEIDENHANSL, Risø National Laboratory.

**COLLEGE 5:  
CRYSTAL AND MAGNETIC STRUCTURES**

"Heavy fermion superconductors".

H. CAPELLMANN, I.L.L.

"The MKD system for crystallographic computing".

P. WOLFERS, Laboratoire de Cristallographie, CNRS, Grenoble.

"Absolute accurate determination of the incoherent X-ray scattering factor  $S(k)$  for  $N_2$  by use of electron spectroscopy".

R.A. BONHAM, Bloomington, Indiana.

"The Structure and dynamics of graphite intercalation compounds".

J.W. WHITE, Oxford P.C.L.

"Neutron texture analysis: comparison of conventional geometry, position sensitive detectors and TOP".

H.R. WENK, University of California.

"Phase transitions in two-dimensional lattices of superconducting vortices".

H. BECK, Université de Neuchâtel.

"The dynamics of charge density waves".

G. GRÜNER, UCLA Department of Physics and Max-Planck-Institut für Festkörperforschung.

"Neutron optics: A brief review of recent experiments and planned projects testing fundamental propositions in quantum mechanics".

A.G. KLEIN, Melbourne.

"Current research in the phase transition kinetics laboratory, Institute of Physics, Jagellonian University".

J. SŁOTYS, Jagellonian University, Krakow.

"Formation and coupling of magnetic moments in Heusler alloys".

C.B. SOMMERS, Orsay.

**COLLEGE 6:  
LIQUIDS, DISORDER AND DEFECTS IN MATERIALS**

"Inelastic neutron scattering from low frequency excitations in glasses".

U. BUCHENAU, Institut für Festkörperforschung, Jülich.

"Random alloys: from Schrödinger's equation to the rolling mill - photoemission studies of their electronic band structure and magnetic properties".

R.G. JORDAN, University of Birmingham.

"Nucleation of intergranular creep cavities".

M.H. YOO, Metals and Ceramics Division, Oak Ridge National Laboratory.

"Microstructural information in solids from channeling experiments".

Y. QUERE, CEN Fontenay-aux-Roses.

"The hexatic phase: synchrotron X-ray scattering studies of liquid crystal thin films".

D.E. MONCTON, Brookhaven National Laboratory.

"The liquid-vapour critical point phase transition of metals".

F. HENSEL, Institute of Physical Chemistry, Philipps-University of Marburg.

"Neutron scattering from solutions: elucidation of hydration properties".

R. HAHN, Transuranium Research Laboratory, Oak Ridge.

"SANS study of grain boundary cavitation".

J.R. WEERTMAN, North Western University, Illinois.

"Time-of-flight experiment with conduction electrons - an application of weak localization".

G. BERGMANN, KFA Jülich.

"Can deuterium-NMR measure an elastic incoherent structure factor similar to quasi-elastic neutron scattering" ?

F. FUJARA, Universität Mainz.

"Hydrogen in amorphous metals".

R. KIRCHHEIM, MPI Stuttgart.

**COLLEGE 8: BIOCHEMISTRY**

"New methods to study viral assembly in the cell".

M. DUBOIS-DALCO, N.I.H. Bethesda.

"Quasi elastic light scattering and NMR self diffusion of aqueous bile salts lecithin solution".

P. SCHURTENBERGER, ETH Zürich.

"ATP requirement for lipid asymmetry in erythrocyte membranes".

Ph. DEVAUX, Institut de Biologie, Paris.

"Structure et polymorphisme des lipides bipolaires d'archaebactéries thermo-acidophiles".

V. LUZZATI, Gif-sur-Yvette.

"Génétique de l'adénovirus : assemblage et réplication".

J.C. D'HALUIN, Lille.

"Régulation de l'expression des gènes des aminoacides tRNA synthetases".

M. GRUNBERG-MANAGO, Institut de Biologie Physico-Chimique, Paris.

"Cell specific expression of cloned genes".

R. CORTESE, EMBL Heidelberg.

"Recent developments in electron microscopy of fibres, sheets and crystals (e.g. microtubules, S-hemoglobin)".

S.J. EDELSTEIN, Cornell, New York.

"Reconstitution of the crystalline purple membrane from purified fragments of bacteriorhodopsin".

M.J.L. POPOT, Institut de Biologie Physico-Chimique, Paris.

"Immortalisation des lymphocytes par le virus d'Epstein-Barr".

J.M. SEIGNEURIN, Laboratoire de Virologie, Faculté de Médecine, Grenoble.

"Biological applications of synchrotron radiation".

S. DONIACH, Université de Paris-Orsay, Laboratoire Léon Brillouin-Saclay and Stanford University.

"Optical methods for the analysis of DNA dynamics".

J. LANGOWSKI, Hannover.

"Transducin and the amplifying cascade of vision".

M. CHABRE, CENG

"Structural study of trypanosome variant surface glycoprotein".

P. METCALF, Harvard University

"The role of elongation factors for the speed and accuracy of the ribosomal elongation cycle, experiments and computer simulations".

F.U. GAST, Hannover.

#### COLLEGE 9: CHEMISTRY

"Neutron scattering studies of micelles and microemulsions".

J. TABONY, Département de Physico-Chimie, C.E.N. Saclay.

"Critical reflection of polarized neutrons".

J.A.C. BLAND, I.L.L.

"The structure and dynamics of graphite intercalation compounds".

J.W. WHITE, Oxford P.C.L.

"Empirical relationship between ionic radii, bond length, bond strength and bond energy".

J. ZIOLKOWSKI, Cracovie.

"Présentation de la diffusion quasi élastique de la lumière (DQL)".

G. LARCHERES, Centre de Recherche de Décines - Rhône Poulenc Recherches.

"Caractérisation physico-chimique des solides divisés".

J.C. LE THIESSE, Centre de Recherche de Décines - Rhône Poulenc Recherches.

"Forming, drying and sintering pure silica monoliths by the sol-gel process".

L.C. KLEIN, Ceramics Department, Rutgers, the State University of New Jersey, Piscataway.

"Robust equipment for dynamic neutron fluoroscopy".

S.J. COCKING, Materials Physics Division, AERE Harwell.

"Concentrated micellar systems: neutron scattering at small and medium momentum transfer".

T. ZEMB, CEN Saclay.

"Phonon scattering in atomically rough surfaces".

A. KHATER, Orsay.

"Branched polymers and gels".

M. DAOUD, Laboratoire Léon Brillouin, CEN Saclay.

"Diffusive motions in polymer molecules on universal and local scale".

B. STUHN, Universität Freiburg.

#### OTHERS

"ESRF and ILL: A European interdisciplinary research centre in Grenoble"?

"Recent advances in solid state and surface spectroscopies".

M. CAMPAGNA, Institut für Festkörperforschung, Jülich.

"Point-contact spectroscopy".

P. WYDER, Max-Planck-Institut für Festkörperforschung, Hochfeld-Magnetlabor, Grenoble.

## **I.L.L. WORKSHOPS AND CONFERENCES**

### **WORKSHOPS ORGANIZED BY THE I.L.L. IN 1985**

**I.L.L. 17-18 January, 1985.** Workshop on 'Future Plans in Nuclear Spectroscopy at the ILL High Flux Reactor' (organized by F. Hoyler)  
[Presented Viewgraphs: ILL Internal Report 85HO18T]

**I.L.L. 4-9 March, 1985.** Course and Workshop on 'Diffusive Molecular Motion', (organized by J.P. Beaufils, M. Bée).  
[Course: ILL Internal Report 85BE10T]  
[Workshop: (Abstracts) ILL Internal Report 85LE9T]

**I.L.L. 26-29 March, 1985.** International Workshop on 'Interpretation and Uses of Electronic Distributions', (organized by P.J. Becker, P.J. Brown, F.L. Hirshfeld)  
(not to be published)

**Grenoble 8-12 July, 1985.** 'Third International Conference on the Grenoble Structure of Non-Crystalline Materials', (organized by Chr. Janot, A.F. Wright)  
Proceedings to be published in *Journal de Physique - Colloques*;  
[Abstracts: ILL Internal Report 85IL20T]

**I.L.L. 15-16 July, 1985.** 'Beyond Radial Distribution' - International Workshop on Higher Order Correlation Functions, and Bond-Angle Distribution in Non-Crystalline Solids and Liquids, (organized by J.-B. Suck, D. Quitmann)  
Proceedings to be published in *Journal de Physique - Colloques*;  
[Abstracts: ILL Internal Report 85IN22T]

**Jülich, W. Germany 2-4 October, 1985.** Workshop on 'Atomic Transport and Defects in Metals by Neutron Scattering', (organized jointly by the ILL and IFF/Jülich: Ch. Janot, W. Petry, D. Richter, T. Springer)  
[Proceedings to be published in 'Proceedings in Physics', Springer Verlag]

**I.L.L. 15-17 October, 1985.** 'Lectures on Weak Nuclear Interactions', (organized by B. Desplanques, P. Geltenbort, F. Gönnerwein)  
[Presented Viewgraphs: ILL Internal Report 85DE28T]

**I.L.L. 4-6 November, 1985.** International Workshop on 'Evaluation of Single-Crystal Diffraction Data from 2-D Position Sensitive Detectors', (organized by A. Filhol, S. Mason, M. Roth, ILL; A. Lewit-Bentley, G. Bentley, EMBL; G. Bricogne, R. Kahn, LURE).  
[Proceedings to be published in *Journal de Physique - Colloques*]

### **WORKSHOPS SPONSORED BY THE I.L.L. IN 1985**

**Geilo, Norway 8-19 April, 1985.** 'Scaling phenomena in disordered systems', R. Pynn, A. Skjeltorp,  
Proceedings of a NATO ASI  
[To be published NATO ASI Series B vol. 133 Plenum, 1986]

### **PUBLISHED IN 1985:**

**Tenerife, Spain 2-3 April, 1984.** NATO ASI Series E N°92, 'Glass... Current Issues', edited by A.F. Wright, J. Dupuy  
1985 Martinus Nijhoff Publishers

## **THESES**

The experimental work of which was carried out at the I.L.L.:  
**BLOENNIGEN F.** — Inaugural Dissertation zur Erlangung des Doktorgrades der Naturwissenschaften der Justus Liebig Universitaet Giessen, 1985. Praezise Q-beta-wert Messungen der Massenkettens 95 < A < 99 mit einem GE (HP)-Detektor-Teleskop.

**FILHOL A.** — Thèse de Doctorat d'Etat ès Sciences, Université de Bordeaux I, Avril 1985. Evolution comparée en fonction de la température ou de la pression des propriétés physiques et structurales de conducteurs organiques unidimensionnels.

**HAQUE A.M.I.** — Inaugural Dissertation zur Erlangung des Doktorgrades der Mathematisch-Naturwissenschaftlichen Fakultät der Universitaet Koeln, 1985. Kernstrukturuntersuchungen am 178Hf mittels Neutroneninduzierter Gamma- und Elektronenspektroskopie.

**LASSAILLY Y.** — Thèse de Doctorat ès Sciences Physiques, Université Scientifique et Médicale et Institut National Polytechnique de Grenoble, Mai 1985. Etude des composés Kondo Magnétiques TmS et CeIn3.

**LINDLEY E.** — Thesis for the Degree of Doctor of Philosophy Faculty of Science Physics, University of Southampton, March 1985. Neutron Scattering Studies of Transition Metal Alloys.

**LINDNER P.** — Dissertation zur Erlangung des Grades "Doktor der Naturwissenschaften" am Fachbereich Chemie der Johannes-Gutenberg-Universitaet in Mainz Juni, 1985. Scherabhaengige Deformation von Polystyrol in verduennter Loesung.

**MARMEGGI J.C.** — Thèse de Doctorat ès Sciences Physiques, Université Scientifique et Médicale et Institut National Polytechnique de Grenoble, Juillet 1985. Contribution à l'étude de domaines et de modulations structurales par la diffraction d'un faisceau polychromatique.

**RICHTER R.** — Inaugural -Dissertation zur Erlangung des Doktorgrades der Mathematisch-Naturwissenschaftlichen Fakultät der Universitaet zu Koeln, Juni 1984. Untersuchung der Kernstruktur von 179Hf mit Hilfe der Gamma-, Beta- und Teilchenspektroskopie.

**SMITH J.C.** — Thesis for the Degree of Doctor of Philosophy, Birkbeck College, London University, August 1985. Protein Dynamics studied by Inelastic Neutron Scattering.

**STANLEY H.B.** — Thesis for the Degree of Doctor of Philosophy, Faculty of Science Physics, University of Southampton, December 1985. Magnetic Properties of Rare-Earth single Crystals.

## EXPERIMENTS CARRIED OUT AT THE I.L.L.

The following is the list of experiments performed at the I.L.L. up to Oct. 1985 for which experimental reports were received before Jan., 24th 1986. Due to the long reactor shut-down, the number of reports received is reduced. For a detailed study of the results, please refer to "1985. I.L.L. Experimental Reports and Theory College Activities". (Reports marked with \* are not available for publication).

### COLLEGE 3: FUNDAMENTAL AND NUCLEAR PHYSICS

3-01-155	SEE	3-01-165	
3-01-165	Mass, charge and energy correlations	PN 1	
3-01-155	study of $^{237}\text{Np}(n_{th}, f)$ with Lohengrin.		
	BARREAU G., LEROUX B., MUTTERER M., ENGELHARDT D., ASGHAR M.		
3-02-263	E0 transition strengths in $^{124}\text{Te}$ .	PN 2	
	HAMILTON W.D., SUBBER A.		
3-02-266	Electron density at the nuclear site for gold alloys.	PN 2	
	KALVIUS G.M., SCHRECKENBACH K.		
3-02-274	E0 intensity measurements in $^{78}\text{Se}$ .	PN 2	
	HAMILTON W.D., SUBBER A.		
3-02-299	Electric monopole transitions in $^{106}\text{Pd}$ .	PN 2	
	COLVIN G., HOYLER F., ROBINSON S.	PN 4	
3-06-47	Gamma-gamma coincidence and angular correlation measurements on $^{86}\text{Br}$ .	PN 6	
	KOBRAS K., BEVERS DORF G., STOEHLKER U.		
3-06-62	Production rates of $^{75-83}\text{Ga}$ and $^{118-132}\text{In}$ at OSTIS.	PN 6	
	KRATZ K.L., GABELMANN H., HOERNER S., PFEIFFER B.		
3-06-63	Qbeta measurements of highly unstable daughters of Rb-fission products.	PN 6	
	BLOENNIGEN F., PFEIFFER B., LIPPERT W., STOEHLKER U., WOLLNIK H.		
3-07-7	Beta-decay asymmetry of the neutron and $g_A/g_V$ .	SN 7	
	BOPP B., DUBBERS D., HORNIG L., KLEMT E., LAST J., FREEDMAN S.J., SCHAERPF O.		
3-08-2	A detailed investigation of the $^{229}\text{Th}(n_{th}, f)$ reaction with Cosei Fan Tutte.	PN 8	
	SICRE A., LEROUX B., ASGHAR M., GOENNENWEIN F.		
*3-08-7	Study of proton even-odd effect in $(n_{th}, f)$ for $^{229}\text{Th}$ and $^{238}\text{Pu}$ with a $\delta E-E$ gas ionisation chamber.	SP, BE	
	ASGHAR M., DJEBARA M., NIFENECKER H., BOCQUET J.P., CRANCON J., MAUREL M., RISTORI C.	PN 8	
3-08-16	Detailed study of the mass- and energy characteristics of the $^{241}\text{Pu}(n_{th}, f)$ fragments.	PN 8	
	WAGEMANS C., SCHILLEBEECKX P., GELTENBORT P., GOENNENWEIN F., OED A.		
3-13-50	Preparation of microporous membranes by heavy ions irradiation of polymeric films.	H22 D	
	PINERI M., BALANZAT E.		
3-13-75	Systematic study of $(n, \alpha)$ and $(n, f)$ -reactions.	H22 D	
	WAGEMANS C., SCHILLEBEECKX P., D'HONDT P., ENSALLEN A., BOCQUET J.P.		

### COLLEGE 4: STRUCTURAL AND MAGNETIC EXCITATIONS

4-01-262	Magneto-elastic interaction in $\text{CeF}_3$ in an external magnetic field.	IN 2	
	SCHAACK G., GEICK R., RAUH H.		
4-01-270	Phonon anomalies of intermediate valent systems at low temperatures.	IN 3	
	SEVERING A., HOLLAND-MORITZ E.		
4-01-275	Inelastic scattering study of Peierls instabilities in $\text{NbSe}_3$ and related compounds.	IN 8	
	MONCEAU P., BERNARD L., CURRAT R., LEVY F., ROUXEL J.		
4-01-279	Investigation of electronic and lattice disorder in $\text{UO}_2$ by observation of the temperature variation of optic phonon modes.	IN 1	
	HUTCHINGS M.T., HAYES W., OSBORN R.		
4-01-292	Incommensurate phase of quartz: 1q phase induced by a uniaxial stress.	D10	
	BASTIE P., BERGE B., DOLINO G., BETHKE J., ZEYEN C.		
4-02-194	Effect of high electric field on the modulated structure in thiourea.	IN 2	
	DURAND D., DENOYER F., CURRAT R., VETTIER C.		
4-02-201	Phonons and electron-phonon-coupling in $\text{Nb}_3\text{Sn}$ .	IN 8	
	GLAESER W., MUELLER P., RENKER B., SCHWEISS P., PINTSCHOVIVUS L.		
4-03-268	Dispersion of magnetic excitations in $\text{CeMg}$ .	IN 2	
	GALERA R.M., PIERRE J.		
4-03-270	Field dependence of the magnetic excitations in neodymium.	IN12	
	MCEWEN K.A., STIRLING W.G., STANLEY H.B.	IN 3	
4-03-273	Diffusive solitons in impure Sine-Gordon chains.	IN11	
	BOUCHER J.P., REGNAULT L.P., MEZEI F.		
4-03-275	Renormalisation with temperature of magnetic excitations in the 2d-XY helimagnet $\text{BaCo}_2(\text{AsO}_4)_2$ .	IN12	
	REGNAULT L.P., ROSSAT-MIGNOD J.		
4-03-279	Stoner excitations and optical magnons in $\text{Ni}_6\text{O}$ .	IN 1	
	PAUL D.MCK., MOOK H.A.		
4-03-314	Longitudinal and transverse solitons in THMC: "Parallel spin component".	IN12	
	BOUCHER J.P., REGNAULT L.P., PYNN R., BOUILLLOT J.		
4-03-326	Magnetic fluctuations in $\text{Ni}_3\text{Al}$ .	IN 8	
	BERNHOFET N.R., LONZARICH G., MITCHELL P.W., PAUL D.MCK.		
4-03-328	Paramagnetic scattering of $\text{Pd}_3\text{Fe}$ .	D 5	
	PAUL D.MCK., HIGGINS S.A., NEAL M.		
4-03-370	Search for crystal field transition in $\text{PuSb}$ at high-energy transfers.	IN 1	
	LANDER G.H., STIRLING W.G., ROSSAT-MIGNOD J., SPIRLET J.C., VOGT O.		
4-04-104	Spin dynamics of intermediate valent systems under pressure.	D 7	
	WALTER U., HOLLAND-MORITZ E.		
4-04-114	Valence fluctuations at high pressures and low temperatures.	IN 6	
	WALTER U., HOLLAND-MORITZ E.		

4-04-127	The effect of pressure on the valence state of Yb-compounds. WALTER U. ,HOLLAND-MORITZ E.	IN 5	5-21-203	Metal exchanged zeolites. ADAMS J.M. ,REES L.V.C.	D 1A
4-04-129	Spin dynamics of some intermediate valent Sm- and Eu- compounds. HOLLAND-MORITZ E.	IN 4	5-21-204	Neutron diffraction studies on synthetic and natural silicates. CHEETHAM A.K. ,THOMAS J.M. EDDY M.M. ,WRIGHT A.F.	D 1A
4-04-134	High resolution investigation of critical dynamics in iron at and below the Curie point. MEZEI F. ,FARAGO B.	IN11	5-21-231	Magnetic and nuclear structure of Sr2Co2O5. OBRADORS X. ,RODRIGUEZ J. COLLOMB A. ,ANNE M.	D 1A
4-04-136	Experimental investigation of three-spin correlations dynamics in ferro-magnets. OKOROKOV A.I. ,GUKASOV A.G. SCHAERPF O.	D17 D 7	5-21-233	Neutron diffraction studies on synthetic and natural silicates. CHEETHAM A.K. ,THOMAS J.M. EDDY M.M. ,WRIGHT A.F.	D 1A
4-05- 11	Local spin fluctuations in PdNi alloys. LINDLEY E.J. RAINFORD B.D.	IN 6	5-21-235	Structural studies of lissicon related solid electrolytes. BRUCE P.G. ,DAVID W.I.F.	D 1A
4-05- 29	Local spin fluctuations in PdNi alloys. LINDLEY E.J. RAINFORD B.D.	IN 5	5-22-220	Prestress induced changes of the Al5 crystal lattice in Nb3Sn multifilamentary wires. FLUEKIGER R. ,GOLDACKER W.	D 1A
4-05- 45	Spin dynamics in alpha-Ce and CePd3. GALERA R.M. ,GIVORD D. ,PIERRE J. SCHWEIZER J. ,VETTIER C.	D 5	*5-22-244	Structural studies of ternary oxide phases. DICKENS P.G. ,CROUCH-BAKER S. WELLER M.T.	D 1A
5-33- 77			5-22-258	Study of structural change during annealing of alkali-doped polyacetylene. AIME J.P. ,DELANNOY P. ,SCHOTT M. POUGET J.P.	D 1B
4-05- 52	Time dependent spin correlation functions in the quasi-1D spin glass FeMgBO4. WIEDENMANN A. ,MEZEI F.	IN11	5-22-264	Structure of LaNi5 deuteride at high temperature. ACHARD J.C. ,LARTIGUE C. PERCHERON-GUEGAN A.	D 1A
4-05- 65	Critical dynamics in Pd 2% Mn alloy. SARKISSIAN B.V. ,RAINFORD B.D.	IN11	5-22-267	Structure determination of the Ca(ND3)6 and Li(ND3)4 compounds. LECLERCQ F. ,DAMAY P.	D 1A
4-05- 81	Magnetic excitations in transition metal glasses. COWLAM N. ,PAUL D.MCK.	IN 2	5-22-270	Size and electronic influences on intermolecular forces of hexafluoromolecular ions. COCKCROFT J.K. ,FENDER B.E.F. FITCH A.N. ,KEARLEY G.J.	D 1A IN10
<b>COLLEGE 5: CRYSTAL AND MAGNETIC STRUCTURES</b>					
5-11-150	Single crystal neutron diffraction structure of Cu (methoxyacetate)2.2H2O at 4K. PROUT C.K.	D15	5-22-276	Martensitic transformation in enstatite (kinetics and structure). SCHRADER H. ,FREY F. ,BOYSEN H.	D 1A D 1B
5-11-152	The low temperature structure of LaD3. SCHAACK G. ,FENDER B.E.F. GREGSON D. ,LOUCA P.	D 15	5-22-TEST	SEE 5-16-TEST	
5-15-228	Study of evolution of hydrogen temperature factor preceding dehydration of Ca(OH)2. BOUILLOT J. ,CHAIX-PLUCHERY O. NIEPCE J.C. ,PANNETIER J.	D 9	5-25-169	Measurement of stress distribution in test samples of 'model' geometry. ALLEN A.J. ,HILL R.I. ,ANDREANI C. HUTCHINGS M.T. ,WINDSOR C.G.	D 1A
5-15-TEST	Thermal vibration in CaSCN below the structural transition at 470K. (Furnace test) MAIR S. ,MCINTYRE G.J.	D 9	5-25-174	Strain in stressed anisotropic polycrystalline materials. ALLEN A.J. ,HUTCHINGS M.T. WINDSOR C.G.	D 1A
5-16-TEST	Study of the cooperative Jahn-Teller transition in TmVO4. BASTIE P. ,SALCE B. , LAJZEROWICZ J.	S 21	5-25-186	Determination of ion exchange velocity of zeolite-Y and alpha-zirconium phosphate by neutron diffraction. KROGH-ANDERSEN E. ,CHRISTENSEN A.N. WANDAHL G.	D 1B
5-16-TEST	Irreversible behaviour in the incommensurate phase of quartz. BASTIE P. ,DOLINO G. , MOGEON M. .	S 21	5-25-187	Solid state preparation and phase transition in the ferroelectric SbTaO4. CHRISTENSEN A.N. ,FJELLVAG H. WANDAHL G. ,PANNETIER J.	D 1B
5-21-187	Structure determination of DO.8ReO3 and related compounds. DICKENS P.G. ,WELLER M.T.	D 1A	5-31-105	Structural and magnetic studies of transition metal salts. BATTLE P.D. ,CHEETHAM A.K. ATTFIELD J.P.	D 1A
5-21-193	Structural determination in LiCl-MCl2 solid solutions (M = Mg, V). CROS C. ,SOUBEYROUX J.L.	D 1B	5-31-128	Origin of the magnetic coercivity in a new important class of permanent magnets. LEMAIRE R. BLEY F. ,LIVET F.	D17
5-21-198	The structure and hydrogen bonding in M3HGe7O16.4H2O. FENDER B.E.F. ,FITCH A.N.	D 1A			

5-31-146	Magnetic order and fluctuations in mica-group minerals. COEY J.M.D. BALLET O.	D17 D'18	5-08-102	Chemical short range order in NiTa metallic glasses. COWLAM N. ,DAVIES H.A. ,GARDNER P.P.	D 4
5-32-174	Magnetic structure determinations of Gd-Y alloy single crystals. PALMER S.B. ,BATES S. MCINTYRE G.J. SOUSA J.B.	D 9	5-08-117	Photostructural changes in thin films. ELLIOTT S.R. ,SPENCE C.A.	D11
5-32-198	Magnetic moment in the helical phase of MnSi. BROWN P.J. ,ZIEBECK K.R.A.	D16	5-08-128	Nucleation and crystallisation in aluminium based amorphous alloys. DUBOIS J.M. (Nancy).	D17
5-32-202	Spin-density waves in Y-Gd. FERT A. ,PUREUR P. CAUDRON B. GIVORD D.	D15	5-08-146	Local order in superionic conductor glasses : AgPO3-MI2 and AgPO3-AgI. MALUGANI J.P. ,TACHEZ M. ,MERCIER R.	D 18
5-32-203	Magnetic structure of single crystal Gd-Y alloys. PALMER S.B. ,BATES S. MCINTYRE G.J.	D 9	5-08-150	Determination of intermediate range order in glassy Si and B chalcogenides. ELLIOTT S.R. ,WRIGHT A.C. SINCLAIR R.N. ,GLADDEN L.F. GRINLEY D.I.	D 4
5-32-206	Neutron diffraction study on the magnetic ordering in beta-EUP3. CHATTOPADHYAY T. BROWN P.J.	D15	5-08-165	The low energy phonon spectrum of epoxy-resins. ROSENBERG H.M. ,PAGE J.N.	IN 6
5-32-TEST	SEE 5-16-TEST		5-12- 2	Small angle scattering of porous silicon. BONCHIL G. ,GOELTZ G. ,BARLA	D11
5-33- 77	SEE 4-05- 45		5-12- 42	Pore size development and gel structure of reactive silica cement systems. ALLEN A.J. ,PEARSON D.	D11
5-33- 97	Bonding in transition element-metalloid alloys and intermetallic compounds. FORSYTH J.B. ,BROWN P.J.	D 3 D 9	5-12- 50	Investigation of anion disorder in KCaF3. BEECH F. ,CATLOW C.R.A. CHADWICK A.V.	D 9
5-33-116	Form factor of CeSn3. BOUCHERLE J.X. ,FLOUQUET F. SCHWEIZER J. ,GIVORD F.	D 3	5-12- 58	Diffuse scattering study of Bi2O3/Yb2O3. CATLOW C.R.A. ,MORONEY L.M. BATTLE P.D.	D 7
5-33-139	A polarised neutron study of spin density in the pseudo-singlet ground state antiferromagnet RbFeCl3. VISSER D. ZIEBECK K.R.A. ,BROWN P.J.	D15 D 3	5-12-TEST	Determination of equilibrium concentration of defects by comparison of macroscopic and microscopic thermal expansion. TROST W. ,MAIER K. ,DIFFER K.	S 21
5-33-149	Spatial distribution of magnetic electrons in PuTe. LANDER G.H. ,DELAPALME A. SPIRLET J.C. ,REBIZANT J. ,VOGT O.	D 3	5-14- 7	Deuterium correlations in VD4 and NbDx. MAGERL A. ,WIPF H. ,BOUILLOT J.	D 18
5-34-114	Effect of applied magnetic fields on the magnetic structure of Gd-Y alloys. PALMER S.B. ,BATES S. SOUSA J.B.	D 5	5-14- 9	Optical vibration modes in Mn and Si substituted LaNi5 hydrides. ACHARD J.C. ,DIANOUX A.J. LARTIGUE C. ,PERCHERON-GUEGAN A.	IN 4
5-34-115	Magnetic form factor of Tm in TmSe. BOUCHERLE J.X. ,BONNET M. FLOUQUET J. ,STUNAUT A.	D 5	5-14- 19	Dynamic of Ag+ ions in a superionic conductor glass AgPO3-AgI. MERCIER R. ,TACHEZ M. ,MALUGANI J.P.	IN 6
*5-34-118	Rotation of neutron polarisation by scattering from Cr2O3. BROWN P.J. ,FORSYTH J.B. ,TASSET F.	D 5	*5-14- 33	Elastic structure factor of tunneling H in NbOxHx, x = 0.002. MAGERL A. ,HEIDEMANN A. ,WIPF H.	IN13
<b>COLLEGE 6:</b>					
<b>LIQUIDS, DISORDER AND DEFECTS IN MATERIALS</b>					
5-03-133	Chemical short range order in CuTi liquid metals. COWLAM N. ,DAVIES H.A. ,CARR G.E.	D 4	5-14- 36	Motion of hydrogen in LaNi5-xMxHy compounds. ACHARD J.C. ,LARTIGUE C. PERCHERON-GUEGAN A.	IN13
5-05- 82	Structure factor of N-methyl-formamide. ZEIDLER M. ,KALMAN E. ,JUNG W. SCHULZ J.	D 4	5-14- 37	Study of a localized jumping mode of hydrogen in beta-LaNi5 and related hydrides. ACHARD J.C. ,DIANOUX A.J. LARTIGUE C. ,PERCHERON-GUEGAN A.	IN 6
5-06- 26	Structure of molten lithium halides. TITMAN J.M. ,COWLAM N. ,CARR G.E.	D 2	5-14- 40	Low and high energy vibration modes for hydrogen in several LaNi5 type hydrides. ACHARD J.C. ,BENHAM M.J. ANDERSON I. ,DIANOUX A.J. LARTIGUE C. ,ROSS D.K. PERCHERON-GUEGAN A.	IN 1
5-06- 37	Neutron diffraction study of liquid Mg.72Zn.28 alloy. ANDONOV P.	D 2	5-14- 45	Energy width of coherent quasielastic scattering from anion disorder in UO2 at high temperatures. HAYES W. ,OSBORN R. ,HUTCHINGS M.T. MACDONALD J.E. ,HACKETT M.A.	IN12
5-07- 63	Structure of ammonium sulphate solution. CUMMINGS S.	D 4			

6-14- 91	Dynamic of Ag <sup>+</sup> ions in two superionic conductor glasses : AgPO <sub>3</sub> -PbI <sub>2</sub> and AgPO <sub>3</sub> -AgBr. MERCIER R. ,MALUGANI J.P. ,TACHEZ M.	IN 5	8-07- 78	Protein-polysaccharide interactions in proteoglycan aggregates. PERKINS S.J. ,HARDINGHAM T.E. MUIR H. ,DUNHAM D.G.	D17
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**COLLEGE 7: IMPERFECTIONS (Now disbanded)**

7-03-238	Chemical decomposition of iron rich iron-chromium alloys. BIGOT J. ,BLEY F. ,LIVET F.	D17
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**COLLEGE 8: BIOCHEMISTRY**

8-02-127	Interaction between tRNA and polypeptide elongation factors under high salt conditions. LEBERMAN R. ,ANTONSSON B. ,JACROT B. ZACCAI G.	D17
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8-03- 82	Structure of 40 S nuclear ribonucleo-protein particles. SCHAEFER K.P. ,WILK H.E.	D11
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8-03- 87	Neutron scattering studies of chromatin structure. BALDWIN J.P. ,WOOD M.J. STAYNOV D.Z.	D11
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8-03- 91	Conformational changes of RNA polymerase during different steps of transcription. HEUMANN H. ,LEDERER H. ,MAY R.P.	D11
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8-03-100	Ni <sup>2+</sup> - A.T.P. ENDERBY J.E. ,JOVIN T. SOUMPASIS D.M. ,CUMMINGS S.	D 4
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8-04- 75	Molecular dynamics of myoglobin and haemoglobin. MIDDENDORF H.D. ,PERUTZ M.F. TORBET J. ,WORCESTER D.L.	IN 6
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8-04- 78	Molecular dynamics of enzymes. MIDDENDORF H.D. ,RANDALL J.	IN 6
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8-04- 85	Intramolecular dynamics of bovine pancreatic trypsin inhibitor(BPTI) studied by inelastic neutron scattering. SMITH J. ,CUSACK S. ,FINNEY J.	IN 5 IN 6
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8-04- 89	Low-frequency modes in mesophilic and thermophilic enzymes. HINZ H.J. ,MIDDENDORF H.D.	IN 6
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8-06- 53	DNA packaging in isometric viruses investigated by neutron scattering from shear-oriented solutions. FEIGIN L. ,OBERTHUER R.C. TIMMINS P.A. ,LVOV Y. ,LINDNER P.	D17
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8-07- 49	Neutron scattering on first component C1 of human complement. PERKINS S.J. ,VILLIERS C.L.	D11
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8-07- 63	ARLAUD G.J. ,BOYD J. ,BURTON D.R. COLOMB M.G. ,DWEK R.A.	
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8-07- 69	Spatial arrangement of structural domains in tryptophan synthase. KIRSCHNER K. ,IBEL K. ,MAY R.P.	D11
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8-07- 72	Definition of mass per unit length and cross-bridge extension in a new homogeneous preparation of myosin filaments. ROWE A.J.	D11
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8-07- 74	Component C3 of human complement and its control proteins and homologues. PERKINS S.J. ,SIM R.B.	D17
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8-07- 75	SANS on alpha acid glycoprotein. PERKINS S.J. ,KERCKAERT J.P. LOUCHEUX-LEFEBVRE M.H.	D17 D11
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**COLLEGE 9: CHEMISTRY**

9-03-339	Elastic and quasielastic scattering in the orientationally disordered phase of hexachloroethane. GERLACH P. ,PRANDL W. DORNER B.	IN 2
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9-03-376	Tunnelling by inversion. THOMAS R.K. ,HEIDEMANN A. ANDREWS L. ,BINDER D. ,ROSER S.J.	IN13
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9-03-394	Rotations of hydrogen molecules in solid neon. LANGEL W.	IN 4
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9-03-433	Rotational tunnelling in substituted tetramethylmetal compounds. PRAGER M. ,WEISS A. ,DA ZHANG	IN10
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9-03-436	Rotational tunneling and librations in matrix isolated molecules. PRAGER M. ,LANGEL W.	IN10 IN 5
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9-03-441	Reorientational and librational motions in (NH <sub>3</sub> (CH <sub>2</sub> ) <sub>n</sub> NH <sub>3</sub> ) MnCl <sub>4</sub> layer compounds, n=4,5. SOURISSEAU C. ,CORNU J.C. GUILLAUME F. ,BEE M.	IN 6
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9-04-313	Elucidation of the identity of the hydrogen-bonded species in HUP and AMHUP via their spectrosc. differ. KEARLEY G.J. ,FITCH A.N. FENDER B.E.F.	IN 1
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9-04-338	Determination of the vibrational spectra of some hydrogen insertion compounds. DICKENS P.G. ,CHIPPINDALE A.M. WELLER M.T.	IN 18
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9-04-349	Linear associates of HCN in inert gases. KNOEZINGER E. ,KOLLHOFF H. LANGEL W.	IN 4
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9-04-350	Dynamics of hydrogen bonds in carboxylic acid dimers. ERNST R.R. ,FURRER A. ,HEIDEMANN A. STOECKLI A. ,SCHOENENBERGER C.	IN13
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9-05-358	Study of the structure of steroidal gels. TERECH P. ,VOLINO F. ,RAMASSEUL R.	D17
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9-06-444	High temperature form of hydrogen chemisorbed on platinum. RENOUPREZ A. ,JOBIC H.	IN 1
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9-06-456	Quantized rotation of H <sub>2</sub> sorbed in aluminosilicates. THOMAS R.K. ,HUNT G. ,COLLINSON S.J.	IN 4
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9-06-457	Forces between clay platelets. THOMAS R.K. ,HUMES R.P.	IN 3
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9-06-461	Dynamical behaviour of C <sub>2</sub> H <sub>2</sub> and H <sub>2</sub> molecule adsorbed in zeolite NaA. COHEN DE LARA E. ,KAHN R.	IN 5
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	85F1136	GIBAUD A.	85LA196		85ZU275
	85WR280	GIGNOUX D.	85G1231		85WR280
FLOUQUET J.	85BE160		85G1239		85HA385
	85H0187		85G11086	HECHT A.M.	85HE1095
	85BE220	GILBOY W.B.	85C0129	HEDOUX A.	85BE258
	85J0262	GILLIER-PANDRAUD H.	85L0123	HEIDEMANN A.	85HE1G
	85LA338		85D1224		85HE101
FOERSTER I.	85T1296	GIVORD D.	85F1188		85HE119
	85HA314		85G1194		85AL141
	85F0315		85BR197		85SU356
FONTANA M.P.	85MA135		85GA218		85HA385
FORD G.W.	85F0297		85G1234	HEIM U.	85MC336
	85F0416		85GA343	HEINZL J.	85K0384
FORGAN E.M.	85MC236		85BR375	HEITJANS P.	85BA281
FORSYTH J.B.	85F11022	GIVORD F.	85G1231		85HE1008
FORT D.	85MC236		85G11086		85K01017
	85MC237	GLADDEN L.F.	85GL1107		85AC1106
	85BA387	GOBERT G.	85HA206	HENDRIKX Y.	85AL1003
FOUASSIER M.	85LA290	GODDARD R.	85G0252	HENNION B.	85ES147
FOURNIER J.M.	85BA130	GOENNENWEIN F.	85DE28T		85P0388
	85B0169		85F1127		85KU1014
	85BA242	GOERLITZ C.	85G0354		85G11086
FRANEK F.	85AL326	GOGOL E.P.	85G01013	HENNION M.C.	85BE397
FRANKLYN C.	85H0305	GORIUS M.F.	85AL263	HENRY J.Y.	85R0417
FRANSE J.J.M.	85FR328	GRAAF L.A.DE	85VA198	HENRY Y.	85B0173
	85FR342	GRANIER T.	85GA124	HERBST L.	85HE208
FRASE K.G.	85TH202	GRASSHOFF P.	85HA316		85HE399
FREDO S.	85GA264	GREAVES G.N.	85GL1107	HERBSTEIN F.H.	85HE325
FREEDMAN S.J.	85B0317	GREEN K.	85F121T	HERRMANN P.	85HE177
FREILAENDER P.	85BA281		85F1227	HERTZ H.G.	85BE394
FREUND A.K.	85R013T	GREENE G.L.	85KE318	HESS S.	85HE118
	85FR162		85KE383		85HE215
	85FR170	GREENWOOD R.C.	85SP309	HEWAT A.W.	85IH152
	85FR273	GREGSON D.	85GR257		85PA165
	85FR379	GREMPPEL D.R.	85V1154		85B0184
FRICK B.	85FR178		85FE367		85LA196
	85FR1048	GRIMM H.	85L1164		85GA228
FRIEDRICH H.	85FR162	GRUPP H.	85K01017		85SU247
FRINGS P.H.	85FR328	GUENTHERODT H.J.	85SU352		85TH274
	85FR342	GUIGAY J.P.	85GU330		85CH361
FRIT B.	85LA363	GUIM I.	85BU104	HICTER P.	85MA351
FRUCHART D.	85VE1005		85BU105	HIGGINS J.S.	85HI1044
	85CH1063	GULIK-KRZYWICKI T.	85SA1051		85HI1049
FRUCHART R.	85CH1063	GYORGY E.M.	85KW298		85R01067
FUJARA F.	85HE1008	HACKETT M.A.	85S1028		85T01076
	85K01017		85AN1100		85GA1104
FUZELLIER H.	85BA327	HAEN P.	85H0187		85GA1105
	85R0404	HAESSLIN H.W.	85R11059		85HI1111
GABRYS B.	85GA1105	HAGENMULLER P.	85G11087	HILL R.G.	85HI1111
GAEHLER R.	85HE177	HALL P.G.	85CL1068	HILLER B.	85BL337
GAL J.	85GA264	HALL P.L.	85TU171	HLAWATSCH G.	85EG304
GALERA R.M.	85GA121	HAMEDOUN M.	85W11025	HOECKER H.	85RA398
	85F1188	HAN C.	85HA385	HOFFMANN H.	85HE208
	85GA218	HANNON A.C.	85WR1108		85HE399
	85GA221	HAQUE A.M.I.	85T1296	HOFMEYR C.	85H0305
	85B0241		85HA314	HOLLAND-MORITZ E.	85WA1043
	85GA343		85F0315		85H01085
GALLET F.	85W0246	HARADA I.	85TA145	HOLLOWAY J.H.	85DE319
GALLOIS B.	85GA124	HARDCASTLE S.E.	85ZA403	HOLTZBERG F.	85H0187
	85GA279	HARRISON W.T.A.	85BA1050	HONG M.	85KW298
GALLOT Y.	85MA291	HARSHMAN D.R.	85AN396	HOPPE R.	85IH152
GANAZZOLI F.	85GA1104	HASCHKE J.M.	85BA242	HORNIG L.	85B0317
GARNIER P.	85B0184			HORSEWILL A.J.	85HE101
GATTI M.	85FA333			HOWARD C.J.	85NE289
					85NE350

HOWARD J.	85EC386	KERR S.A.	85KR217	LASSAILLY Y.	85H0187
HOWARD J.A.K.	85GR257		85B0295		85L0232
HOWE M.A.	85MC1070		85T1296		85LA250
HOWE R.A.	85NE1071		85EG304		85LA301
HOYLER F.	85H018T		85B0306		85LA338
	85B0295		85B0307	LASSEGUES J.C.	85LA290
	85B0306		85SC310		85DA1036
	85SC310		85KE312	LAST J.	85B0317
	85H0311		85HA314	LAUGIER J.	85JA269
	85KE312		85F0315	LAUNAY J.M.	85LA196
	85HA316	KESSLER E.G.	85KE318	LAUPPE W.	85BR1015
	85EG390		85KE383	LAUTER H.J.	85C0159
HUANG C.Y.	85AN396	KEYSER U.	85KE1090		85WA182
HUBER K.	85HU278	KHAMIS H.W.	85P125T		85R0405
HUEBSCH J.	85VE1005	KIESE G.	85BA281	LAVAL J.P.	85LA363
HUNGERFORD P.	85KR217	KILVINGTON A.I.	85AG216	LAVAL J.Y.	85LA196
	85T1296	KIRSCHNER K.	85B331	LAWIN H.	85ME226
	85EG304	KISCHKO U.	85B0169		85MU256
	85B0307		85FR170	LE BAIL A.	85LE1082
	85SC310		85B0346		85LE1083
HUNTLEY J.M.	85BR265		85K1368	LE CAER G.	85VE1005
HUTCHINGS M.T.	85OS1028	KLAPDOR H.V.	85HA316	LEADBETTER A.J.	85LE9T
	85AL1060	KLEMT E.	85B0317		85LE1052
	85AN1100	KLENZE R.	85LA110	LEBLANC M.	85FE134
IBEL K.	85ST140	KNOEZINGER E.	85LA366		85FE151
	85KN156	KNOLL W.	85KN156		85FE271
	85KN157		85KN157		85K0277
	85HE208		85KN320		85LE378
	85LE245	KNOPP G.	85KN345	LECHNER M.D.	85LE245
	85KN320	KNORR K.	85L0181	LECHNER R.E.	85LE1037
	85B331		85KN345	LECLERCQ F.	85LE1041
	85B365	KOANG D.H.	85AV1030	LECOMTE M.	85PL1032
	85HE399	KOBERSTEIN J.T.	85K01038		85PL1033
IDRISSI A.	85AV1030	KOCH H.R.	85MC336	LEFEBVRE J.M.	85LE1094
IHLE G.	85IH321	KOEHLER W.C.	85BA387	LEGRAND E.	85HA206
IHRINGER J.	85IH152	KOERBER A.	85HA316	LEGVOLD S.	85BA387
INOUE H.	85IN382	KOERBLEIN A.	85HE1008	LEHMANN M.S.	85LE11T
ISHIKAWA Y.	85PA254		85K01017		85L0123
	85GI1086	KOLLHOFF H.	85LA366		85PU125
IZAK-BIRAN T.	85BR1015	KONDUROV I.A.	85B0307		85LE137
JACOBONI C.	85LE1082		85MC336		85LE209
	85LE1083	KORSCHINEK G.	85K0384		85SC214
JAL J.F.	85JA143	KOZAK A.DE	85FE151		85HE325
	85A0419	KRESS R.B.	85HE325		85CH332
JANOT C.	85JA20T	KRUEGER C.	85G0252		85LE340
	85JA111	KRUSCHE B.	85KR217		85LA369
	85JA269		85EG304	LEITNER W.	85HA316
	85JA364	KUEGLER K.J.	85KU1012	LEMAIRE R.	85GI231
JENSEN A.S.	85DU322	KUENZI H.U.	85SU356		85GI239
JEX H.	85L0181	KUGEL G.	85KU1014		85GI1086
JOBIC H.	85F1136	KUHS W.F.	85BA130	LETOFFE J.M.	85A0374
	85BR153		85NE199	LEWIS J.T.	85F0416
	85BE341		85NE289	LHERSONNEAU G.	85ME226
	85S0400		85NE350	LI H.S.	85GI194
	85RE401	KURTZ W.	85KU1077	LIEB K.P.	85KR217
	85J01054	KWO J.	85KW298		85EG304
	85J01101	LAARIF A.	85TH274	LIENARD A.	85GI234
JOHNSON S.M.	85CA179	LACOUT J.L.	85SU247		85BA270
	85BR197	LALIGANT Y.	85LA16T	LILES C.	85L115T
	85J0262	LAMBERT B.	85AL263	LINDLEY E.J.	85N0358
JOHNSON W.L.	85MA353	LAMPARTER P.	85WI1021	LINDNER P.	85L1253
	85WR1108	LANCERI L.	85FI21T	LINK K.H.	85L1164
KAERTS E.	85KE312		85FI227	LITTERST F.J.	85GA264
KALUS J.	85HE208	LANDER G.H.	85MU102	LOCKE J.	85L01034
	85HE399		85LA110	LOEWENHAUPT M.	85FR178
KALVIUS G.M.	85GA264		85LA161		85FR1048
KAMBOUR R.P.	85MA1010		85LA191		85L01084
KAMITAKAHARA W.A.	85ZA403	LANDESMAN J.P.	85LA1109	LOGINOV YU.E.	85B0307
KANE W.	85B0307		85LA122		85MC336
	85SC310		85CH166	LOIDL A.	85L0181
	85MC336	LANE A.N.	85B331	LONGCHAMBON F.	85L0123
KANELLAKOPOULOS B.	85LA110	LANGEL W.	85LA366	LOONG C.K.	85L01084
KANNO R.	85GI1087	LAPIERRE F.	85H0187	LOPES L.C.	85L0232
	85S01088	LAPP A.	85NI158	LORENZELLI N.	85LA122
KAPON M.	85HE325		85NI391	LOUCHEUX-LEFEBVRE M.	85PE1066
KEARLEY G.J.	85KE133	LAPUJOULADE J.	85LA1110	LUCHELLI E.	85GA1104
KEITEL R.	85AN396	LARSEN S.	85VI154	LUSHINGTON K.J.	85HE119
KELLER-BESREST F.	85PL1033	LARTIGUE C.	85LA369	LUTZ P.	85HU278
KERCKAERT J.P.	85PE1066	LARYSZ J.R.	85LA395	LYNN J.W.	85BR197
			85GE283	MACDONALD J.E.	85OS1028
				MACMAHON T.D.	85MC336
				MACONNACHIE A.	85TA145
					85MA1010

MADIH K.	85C0159	MEZEI F.	85ME142	OBERTHUER R.C.	85N1158
MAGERL A.	85HE1G		85ME167		85LE245
	85CA109		85SH190		85L1253
	85MA255		85AL326		85B365
	85BA327		85GU330		85N1391
	85MA357		85B0334		85RA398
	85ZA403	MIGLIARDO P.	85MA135		85RE401
	85R0404	MIKOU A.	85LA363	OED A.	85F1127
MAGNANI M.	85AB349	MILLER G.G.	85A11026	OKPALUGO D.E.	85OK186
MAIER B.	85JA20T		85A11069		85OK1018
	85DE28T	MIRANDA A.	85DU322		85DU322
MAIER H.J.	85K0384	MIRANDA P.C.	85MI410	OLIVIERI C.A.	85F11056
MAISANO G.	85MA135	MITCHELL E.W.J.	85MC1016	ORR G.B.	85MC336
MALAMAN B.	85VE1005		85L01034	OSBORN R.	85OS1028
MALETTA H.	85SH190		85MC1070		85AN1100
MAMPE W.	85F121T	MITCHELL P.W.	85MI292	OSTANEVICH Y.M.	85AL326
	85HE177	MITSCHLER A.	85L0123	OTTEWILL R.H.	85HA268
	85AG216	MITSUNARI T.	85MC336	PAAR V.	85T0225
	85F1227	MIURA S.S.	85ER183	PALLESCHI V.	85FA333
	85GE283	MOLINARI A.	85ST248	PALMER S.B.	85PA254
MANES L.	85B0169	MONNAND E.	85ME226		85BA387
	85BA242		85MU256	PANKOVE J.I.	85CH412
MANGIN P.	85TE115	MONTAGUE D.G.	85M0113	PANNETIER J.	85FE134
	85TE294	MOOK H.A.	85M0176		85CR146
	85R0339	MOORE M.W.	85WI148		85FE151
	85MA389		85DA229		85SC214
MARCHAL G.	85TE115		85DA409		85AL263
	85TE294		85M01058		85FE271
	85JA364	MORE M.	85AM259		85RE276
MARCHIORO A.	85F121T	MOREAU J.M.	85G1239		85K0277
	85F1227	MORENO M.	85R0417		85LA363
MARCUS J.	85ES147	MORET R.	85P0388		85CH370
	85P0388	MORIN P.	85M0106		85LE378
MARET M.	85YA260	MORITZ K.	85KU1012		85B1392
	85MA353	MORRISON J.A.	85HE119	PAPE R.DE	85LE378
MARGACA F.M.A.	85MC1070	MOUDDEN A.H.	85P0388	PATTERSON C.	85PA254
MARIE P.	85MA291	MOZE O.	85M0358	PAUL D.MCK.	85M0176
MARK F.	85G0252	MUELLER-WARMUTH M.	85PR1006		85M1292
MARMEGGI J.C.	85DE207	MUELLNER M.	85L0181		85M0358
MARTIN A.	85MA117	MUENZEL J.	85MU256	PAUL I.C.	85HE325
MARTINS A.F.	85D1224	MURANI A.P.	85MU102	PAUL W.	85KU1012
MARTYNOV V.V.	85B0307		85MU108	PAWLEY G.S.	85PA165
MASON R.	85F11022		85GA121	PEISL J.	85BE14T
MASON S.A.	85ER183		85MU155		85D0285
	85GR257		85FR178		85D01096
	85LE340		85GA218	PENDLEBURY J.M.	85F121T
MASSOUMI G.R.	85MC336		85MU219		85F1227
MATTAUSCH H.J.	85MA407		85GA221	PENFOLD J.	85HA175
	85UE408		85GA343		85BU1091
MATTENS W.C.M.	85MU102		85KN345	PERCHERON-GUEGAN A.	85LA395
MATTIOLI F.	85F121T	NEILSON G.W.	85C0282	PERKINS S.J.	85PE1064
	85F1227		85NE1114		85PE1065
MAY R.P.	85MA4T	NELMES R.J.	85NE199		85PE1066
	85MA5T		85NE289	PERRICHON V.	85J01054
	85B331		85NE350	PERRIN M.	85DE207
MCDONALD J.E.	85AN1100	NELSON R.	85AN8T	PETRICEK V.	85PE293
MCDONALD P.J.	85HE101	NEUMAIER K.	85HE101	PETRY W.	85V0287
MCEWEN K.A.	85ST192		85HE119	PFEIFFER B.	85ME226
	85MC236	NEUMANN D.A.	85ZA403		85MU256
	85MC237	NEWPORT R.J.	85NE1071	PHAM-THI M.	85PH1072
MCGREEVY R.L.	85DA1007	NGOC THUAN N.	85L0232		85PH1113
	85MC1016	NGUYEN TRUNG C.	85NG201	PICOT C.	85K01038
	85L01034	NIELSEN M.	85CH332		85LE1094
	85MC1070	NIERHAUS K.H.	85ST140		85LA1110
MCINTYRE G.J.	85L0181	NIERLICH M.	85N1158	PIDDUCK A.J.	85CL1068
	85TH202		85N1391	PIECUCH M.	85JA364
	85ST205	NISTLER W.	85FR162	PIERRE J.	85GA121
	85SC213	NOAKES D.R.	85AN396		85F1188
	85LE340	NOLTE E.	85K0384		85GA218
	85BA387	NOUET J.	85LA196		85GA221
MCWHAN D.B.	85KW298	NOVAK A.	85PH1072		85B0241
MELAMUD M.	85GA264	NOVION C.H.DE	85LA122		85GA343
MELONE S.	85B0163	NOWAK A.K.	85WR1102	PILOTTI M.U.	85P125T
	85AB349	NOZIERES P.	85UW107	PINSTON J.A.	85ME226
	85G1239		85N0112	PINTO H.	85GA264
MENDIA-MONTERROSO R.	85BE411		85N0200	PINTSCHOVIVUS L.	85RE138
MENELLE A.	85R11059		85W0246		85P11023
MENKE K.	85FR342	NUNN S.	85WR185	PIRNER H.J.	85IH321
MENOVSKY A.	85GA279	NYSTROM B.	85R01067	PLUMIER R.	85PL1020
MERESSE A.	85AL1009	O'CONNELL R.F.	85F0416		85PL1032
MESSOLORAS S.	85L01034	OADES G.C.	85DU322		85PL1033
	85T0225			POINSIGNON C.	85NG201
MEYER R.A.	85ME226			POLLARD N.J.	85BA1050

POTZEL W.	85GA264	RIVAS E.	85SA1051	SCHMITT-RINK S.	85N0200
POUCHARD M.	85S01088	ROBERT A.	85R027T	SCHNERING H.G.V.	85CH370
POUGET J.P.	85ES147	ROBINSON R.A.	85R013T	SCHOTT M.	85A11026
	85P0388		85R0360		85A11069
POWLES J.G.	85DE319	RODMACQ B.	85R0339	SCHRECKENBACH K.	85C0126
PRAGER M.	85PR1006		85MA389		85T0225
PRANGE R.E.	85FE367	RODRIGUEZ F.	85R0417		85GE283
PRASSIDES K.	85PR1027	ROHWER T.	85HA316		85B0295
	85PR1099	ROLLEY E.	85W0246		85T1296
PRESS W.	85HE119	ROOMS G.	85HA206		85C0303
PROSPER H.B.	85F121T	ROOTS J.E.	85H11044		85EG304
	85F122T		85R01067		85H0305
	85PR414		85GA1104		85B0306
	85PR415	ROQUES B.	85VE1005		85B0307
PUGLIERIN G.	85F121T	ROSENBERG H.M.	85R01103		85ST308
	85F122T	ROSENMAN I.	85BA327		85SP309
PULITI P.	85B0163		85R0404		85SC310
PUREUR P.	85BR375		85R0405		85H0311
PUSEY P.N.	85HA268	ROSS D.K.	85TU171		85KE312
PUSHKAREV V.V.	85CH288	ROSSAT-MIGNOD J.	85B0173		85GE313
PUTKONEN M.L.	85PU125		85R0243		85HA314
PYNN R.	85PY114		85R0344	SCHRECKENBACH K.	85F0315
	85HE118		85W11025		85SC323
	85CH288	ROSSET R.	85BE397		85C0335
	85R0360	ROSTAMI S.	85MA1010		85MC336
QUENTIN P.	85BA150	ROTH M.	85AL180	SCHUCK P.	85HA174
QUITMANN D.	85QU22T		85R0299		85ST248
RABENSTEIN D.	85MC336		85JA364		85BL337
RAE SMITH A.R.	85CH1061		85CH412		85HA347
RAGNARSSON I.	85ME226	ROTH S.	85R11059		85SU362
RAGNETTI M.	85RA398	ROULT G.	85LA363	SCHUETZE H.	85HA402
RAINFORD B.D.	85ST235	ROUX M.T.	85AL263	SCHULT O.W.B.	85B0317
	85RA240	ROWE M.	85HA385		85T0225
	85M0358	RUDIN H.	85SU352		85B0295
	85TE1047		85SU356	SCHULZ H.J.	85SC120
RAMASSEUL R.	85MA291	RUEBENBAUER K.	85B1392	SCHUMACHER M.	85SC1073
RAMEAU A.	85BU1091	RUFIN D.	85BE160	SCHUSSLER F.	85ME226
RAMSAY J.D.F.	85HA316	RUMIANTSEV A.YU.	85CH288	SCHWEIZER J.	85BE160
RAPP V.	85T1296	RUPPERSBERG H.	85G0354		85F1188
RASCHER R.	85HA314	RUSH J.J.	85EC386		85BE220
	85F0315	RUSTICHELLI F.	85BA130		85B0233
	85RA376		85B0163		85G1234
RAUCH H.	85RA1089		85B0169		85G1239
	85R0344		85BA242		85DE266
RAVOT D.	85GL1107		85AB349		85BA270
RAYMENT T.	85LA110	RYAN T.W.	85NE289		85GA343
REBIZANT J.	85BA130		85NE350		85B0346
	85LA191	SACKMANN E.	85KN320		85R0405
	85BA242	SADLER D.M.	85SA1051	SEEGER A.	85HA206
REBOUILLAT J.P.	85G1234		85SA1053	SELB J.	85MA291
	85BA270	SAINT-JAMES R.	85PL1032	SENATEUR J.P.	85CH1063
REES B.	85L0123	SAKURAI J.	85BE220	SENBA M.	85AN396
REGNAULT L.P.	85B0173	SAMOUEL M.	85FE151	SEO T.	85ME226
	85B0334	SARKISSIAN B.V.B.	85K0277	SEYFARTH H.	85T0225
	85R0344		85SA189		85B0295
REICH C.W.	85SP309		85RA240		85B0306
REICHARDT W.	85RE138	SAUVAJOL J.L.	85AN396		85B0307
REISNER G.M.	85HE325	SCHAERPF O.	85SA406	SHAKED H.	85MC336
REISS-HUSSON F.	85SA1051		85BE258	SHAPIRO S.M.	85GA264
REIZLEIN K.	85HE399	SCHALT W.	85SC203	SHARMAN P.	85SH190
RENARD J.P.	85B0334	SCHAUPERT K.	85B0317		85F121T
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