

# ANNUAL REPORT 87

INSTITUT MAX VON LAUE-PAUL LANGEVIN-GRENOBLE-FRANCE

# 20



On  
19 January  
1987  
the Institut  
celebrated  
the 20 th  
anniversary  
of its  
foundation.



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INSTITUT MAX VON LAUE-PAUL LANGEVIN-GRENOBLE-FRANCE

# 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 H. Blank

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

76 48 70 82 F. D. Dijoux (Secretary)

Telex: 320621 F

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

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

The ILL makes a limited contribution towards the travel and subsistence expenses for experimentalists coming from approved laboratories in the four 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 June 1986.

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

Brochure "20 years ILL" issued on the occasion of the 20th anniversary of the ILL on 19 January 1987

all available from SCAPRO

Experimental Reports and Theory College Activities 1987 available from the ILL Library.

## Front Cover

On 19 January 1987 the ILL celebrated the 20th anniversary of its foundation.

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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 1st, 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 1992 and beyond unless two years' notice of termination has been given.

In December 1986, an agreement on "Scientific Membership" of Spain was signed by the ILL and the Spanish Interministerial Commission on Science and Technology for a period of five years starting on January 1st, 1987\*.

Switzerland will probably become another "associated scientific member" of the Institut in 1988. The pertinent agreement between the ILL and the Swiss Bundesrat für Bildung und Wissenschaft is expected to be signed early next year.

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'Énergie 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 service institute created so that chemistry, solid state physics, fundamental and nuclear physics, biology and metallurgy specialists from laboratories in the partner countries can use the unique power of neutron techniques to broaden the attack on their problems. Designing and operating instruments and helping the visiting users to carry out their experiments is thus the principal task of the 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.

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

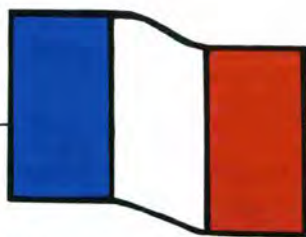
# External Organisation of the Institut Laue-Langevin 1987

## Associates of the Institut

Associated "Scientific Member"



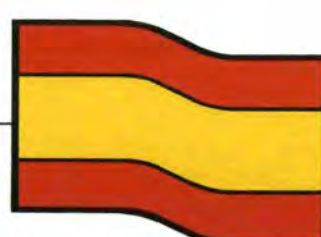
Science and Engineering  
Research Council (SERC)



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



Kernforschungszentrum  
Karlsruhe (KFK)



Comision Interministerial  
de Ciencia y  
Tecnologia

## Steering Committee (at its last meeting)

Atkinson (SERC)	Cribier (CEA)	Blaesing (BMFT)
Clark (SERC)	Fayard (CNRS)	Klose (KFK)
Coles (Imp. Coll. London)	Lehmann (CNRS)	Peisl (Univ. of Munich).
Leadbetter (SERC)	Winter (CEA)	Schunck (BMFT)

## Audit Commission

Badham	Poldermann	Täschner
Middlecote	Metoudi	Riess

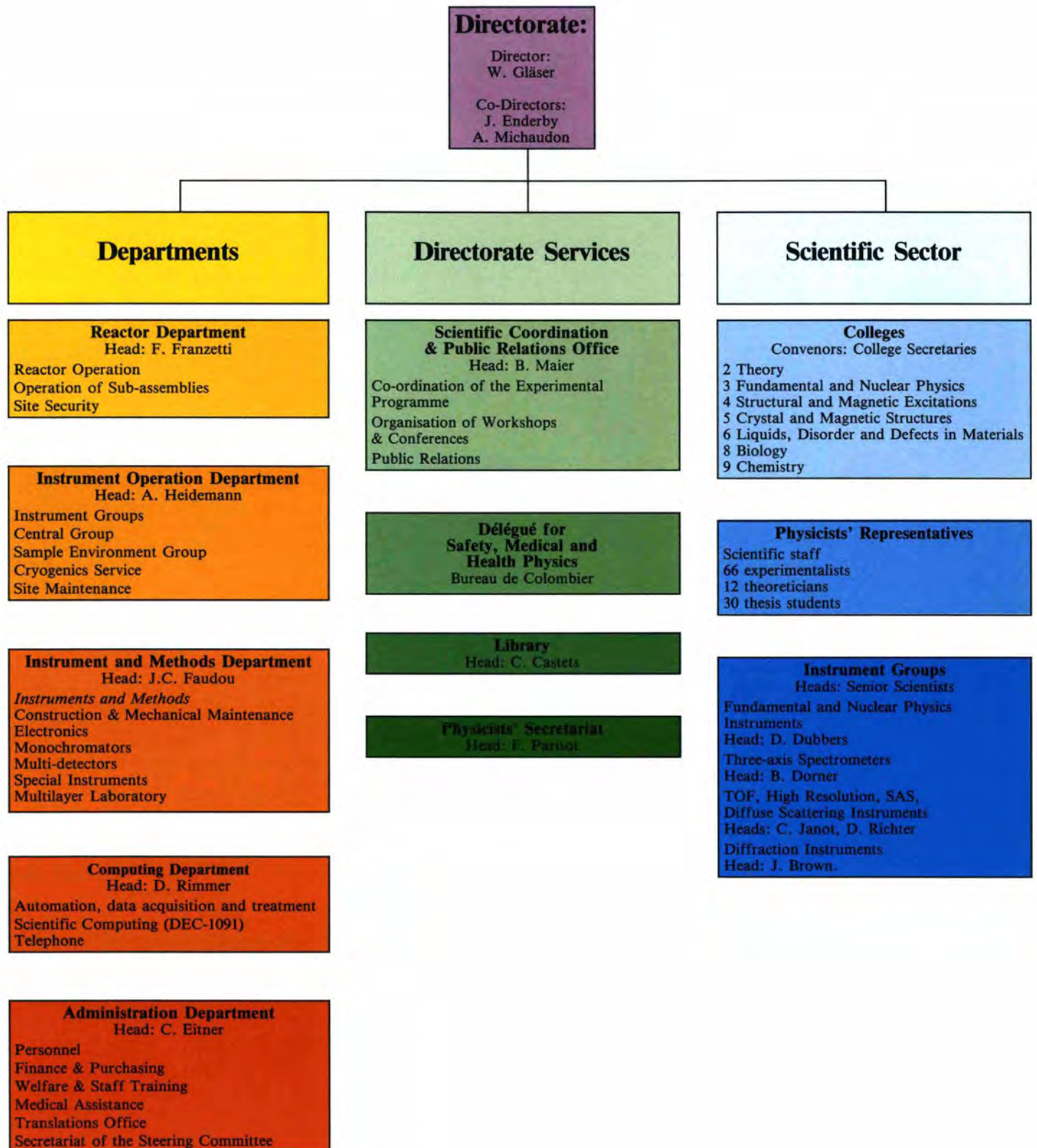
## Scientific Council (at its last meeting)

Barloutaud — CEN Saclay	Gläser — ILL	Moras — CNRS Strasbourg
Blanquet — CNRS Palaiseau	Gomez Sal — Univ. Santander	Petroff — LURE
Buckingham — Univ. Cambridge	Haensel — Observer ESRF	Press — Univ. Kiel
Comes — Orsay	Hensel — Univ. Marburg	Rossat-Mignod — CEN Grenoble
Eitner — ILL	Hoffmann — Univ. Bayreuth	Sackmann — Garching
Enderby — ILL	Howard — ICI	Scherm — PTB Braunschweig
Finney — Univ. London	Kalus — Univ. Bayreuth	Schult — KFA Jülich
Flouquet — CNRS	Lambert — CEN Saclay	Steiner — HMI Berlin
Fuller — Univ. Keele	Maier — ILL	Stewart — Univ. Reading
Gelletly — Univ. Manchester	Mc Ewen — Birkbeck College	Thomas — Univ. Oxford
Geltenbort — ILL	Michaudon — ILL	Vettier — ILL
		Weill — CRM Strasbourg

## Subcommittees of the Scientific Council (at its last meeting)

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	
Barloutaud	Boucher	Ferey	Babel	Comes	Bayley	Batallan	Hoffmann	Higgins
Barreau	Hennion	Finney	Bourée	Franck	Blanquet	Beaufils	Jannink	Leroux
Gelletly	Löwenhaupt	Heger	Burlet	Gaskell	Bode	Howard	Ottewill	Newport
Lieb	Mitchell	Howard	Cheetham	Gerl	Finch	Kettle	Picot	Papoular
Metag	Paul	Jacobs	Flouquet	Neilson	Hinz	Lahrer	Rance	Reichardt
Pendlebury	Reichardt	Raveau	Forgan	Peisl	Jacrot	Schwentner	Strobl	Roudaut
	Squires		Györffy	Sinclair	Moras	Wagner		Scherm
	Steiner		Maletta	Steeb	Philipson			Steichele
	Vallade		Obradors	Teixeira				Taylor

# General Organigramme



## The 20 years of the ILL - 1967-1987

On 19 January 1967 the intergovernmental agreement for the creation of the ILL had been signed by France and the Federal Republic of Germany.

To mark this event, a ceremony was organized at the ILL on Monday, 19 January 1987. The Institute had pleasure in receiving 70 invited guests and dignitaries who celebrated the event together with the ILL staff.

Professor W. GLÄSER, Director of the ILL, opened the ceremony. His speech was followed by addresses from Mr. A. CARIGNON, Mayor of Grenoble ; Prof. H. MAIER-LEIBNITZ (founder and former Director of the ILL) ; Mr. J. HOROWITZ (former Director of the Institut de Recherche Fondamentale, Commissariat à l'Energie Atomique, France) and Prof. E.W.J. MITCHELL, CBE FRS (President of the Science and Engineering Research Council, U.K.).



Prof. W. Gläser  
(Director of the ILL)

### SPEECH BY W. GLÄSER

C'est un grand honneur pour moi d'inaugurer cette fête à l'occasion du 20<sup>e</sup> Anniversaire de l'Institut Max von Laue - Paul Langevin, et de vous dire chaleureusement que vous êtes les bienvenus.

J'aimerais tout particulièrement saluer les conférenciers de cette journée :

Monsieur Carignon, le Maire de Grenoble.

Monsieur Maier-Leibnitz, le Directeur fondateur de l'ILL.

Monsieur Horowitz qui a contribué de façon décisive à la préparation de la fondation et ensuite a participé activement au développement ultérieur de l'Institut, et enfin Monsieur Mitchell qui a collaboré de façon active à la préparation de l'adhésion du Royaume-Uni en 1973.

Le 19 janvier 1967, l'Institut a été créé officiellement par la signature d'un contrat entre les gouvernements de la République Française et de la République Fédérale Allemande. Le but du contrat était de mettre à la disposition de la communauté scientifique des pays membres une source de neutron de très haute intensité pour la recherche dans les domaines de la physique nucléaire, la recherche sur les matériaux, la chimie et la biologie.

Les préparations du côté scientifique ont commencé dès 1964. J'ai eu le privilège, en tant que jeune scientifique, de pouvoir participer très tôt aux discussions préparatoires. J'ai un bon souvenir des réunions à Saclay, au lac de Spitzing, à St-Nizier et à Karlsruhe. Au cours de ces réunions, on avait l'occasion de faire plus ample connaissance avec les collègues des deux pays et de discuter ensemble de la conception du réacteur, de la source froide et des nouveaux instruments. M. Maier-Leibnitz et M. Horowitz peuvent certainement nous en dire plus au sujet de cette période préliminaire.

Je crois que les amitiés qui se sont forgées à cette époque ont contribué de façon significative à la réussite de l'exécution du projet.

Pour la construction de l'Institut, il fallait certainement qu'au début, les deux pays soient tout-à-fait d'accord sur la nécessité de passer au stade de la réalisation le plus rapidement possible, et de définir dès le début les statuts de l'Institut à savoir sa Direction scientifique et son Comité de Direction. Celui-ci a donné les directives générales à l'Institut et a prêté son concours partout où il le pouvait.

Le réacteur a été achevé dès 1971, il a divergé en août 1971 et a atteint sa pleine puissance de 57 MW en décembre de la même année. Dès 1972, il a été possible de commencer l'exécution du programme scientifique. Une large gamme de nouvelles techniques était disponible. Elles avaient d'abord été essayées sur des réacteurs moins grands en Allemagne et en France, comme les guides de neutrons, la technique de diffusion à petits angles et la méthode de "backscattering" à haute résolution, pour n'en citer que quelques-unes. Avec cet équipement, l'ILL a rapidement atteint une position de pointe dans le domaine de la recherche neutronique.

En 1973, le Royaume-Uni a adhéré, comme troisième partenaire, à l'ILL.

Il était important que l'Institut soit conçu dès le début comme un Institut de service, qui mette des équipements expérimentaux uniques à la disposition des chercheurs intéressés des pays membres, et en particulier des groupes universitaires. Lors de la préparation et de l'exécution du programme scientifique, la Direction est conseillée par un Conseil Scientifique. Actuellement, celui-ci dispose de 9 Sous-Comités, dans lesquels est représentée la communauté des utilisateurs, qui sont des chercheurs dans des disciplines très variées. Les sous-comités sélectionnent les meilleures propositions d'expériences soumises.

Après presque 13 ans de fonctionnement impeccable, le réacteur a été révisé dans le cadre d'un important Programme de Modernisation pendant un long arrêt du réacteur. Presque tous les éléments soumis à une irradiation intense ont été remplacés. La source Froide Verticale a été améliorée de façon significative, une série de spectromètres plus performants a été construite et la construction d'une deuxième source froide a été commencée. Depuis son redémarrage en août 1985 le réacteur a marché sans incident. Une étude approfondie de sûreté a conduit à la conclusion que les installations correspondent, sous tous les aspects, aux normes de sécurité. C'est pourquoi j'aimerais, aujourd'hui, mentionner tout particulièrement les différents organismes de sûreté français.

Je voudrais, à cette occasion, souligner leur collaboration efficace et remercier leurs représentants respectifs et plus particulièrement ceux du Service Central de Sûreté des Installations Nucléaires de Base (ceux des Services de Protection et de Sûreté Nucléaire et de la Direction Régionale de l'Industrie et de la Recherche).

Je crois que c'est également une bonne occasion aujourd'hui de remercier l'équipe Réacteur pour son excellent travail pendant toutes ces années. Bien sûr, il faut également remercier tous les autres services techniques ainsi que les scientifiques, puisque sans leur engagement et leurs efforts le Réacteur n'aurait pas pu être exploité avec autant d'efficacité.

L'année 1986 a vu à elle seule la réalisation de 850 expériences. Bien que ce chiffre soit impressionnant, le temps de mesure demandé est toujours 2 ou 3 fois plus grand que le temps disponible. Cette demande continue de croître, par exemple l'année dernière l'Espagne a adhéré à l'ILL et d'autres pays pourraient suivre.

Je suis convaincu que ce bilan positif encouragera les Associés à exploiter l'ILL bien au-delà de l'année 1992.

L'ILL est également au cœur d'une autre initiative européenne dans le domaine de la recherche de la matière condensée. Ainsi l'année dernière cinq pays ont décidé de participer à la construction de la Source Européenne de Radiation Synchrotron sur un site attenant à l'ILL.

Le rayonnement neutronique et synchrotron offre des possibilités complémentaires pour la recherche. Nous croyons que ces possibilités peuvent être utilisées au mieux si les deux installations sont exploitées sur un terrain commun et si l'on profite de toutes les possibilités de collaboration technique et scientifique.

Dans ce sens, j'aimerais encore une fois remercier tous ceux qui voici 20 ans ont créé ici à Grenoble les conditions pour cette collaboration unique et réussie à l'échelle européenne.

## SPEECH BY A. CARIGNON

Je suis très heureux, et très honoré d'être aujourd'hui l'invité de la Communauté Scientifique, à l'occasion du 20<sup>e</sup> Anniversaire de l'Institut Laue-Langevin.

En tant que Maire de cette ville, je voudrais remercier tous ceux qui ont beaucoup travaillé pour créer à Grenoble un centre de recherche de dimension nationale ou internationale.

Tous les 10 ans.

Il y a là, une performance que beaucoup



Monsieur A. Carignon  
(Mayor of Grenoble)

de villes en France et en Europe nous envient.

En effet,

1946 : Création du 1<sup>er</sup> Laboratoire du C.N.R.S. : Mes remerciements vont immédiatement au Professeur Néel, qui, fidèle à Grenoble, nous a encore rendu visite en octobre 1985, mais également au Doyen Weil.

1956 : Naissance du C.E.N.G. : Là encore, hommage rendu à Louis Néel, créateur de cet établissement, aidé dans sa tâche par les patrons du C.E.A., et tout particulièrement Francis Perrin.

1967 : Signature de l'accord créant l'Institut Laue-Langevin, par MM. Stoltenberg et Peyrefitte : c'est à cette date que Grenoble prend réellement une dimension internationale. Certes, il y avait jusqu'alors de nombreux étudiants et stagiaires étrangers dans les laboratoires grenoblois, mais avec la naissance de cet établissement international, les responsables locaux prennent conscience de l'importance des problèmes :

- accueil et intégration des chercheurs et de leurs familles ;
- inscription de leurs enfants dans les établissements scolaires.

A partir de 1967, Grenoble s'équipe et pourra ainsi, 20 ans plus tard, faire face à l'arrivée du Synchrotron : ce projet pour lequel je tiens à remercier tout particulièrement M. Fender, M. Michaudon et bien sûr M. Haensel.

J'allais oublier de souligner l'arrivée en 1976 du C.N.E.T. à Meylan.

A ces événements, je voudrais associer ceux que nous ne connaissons pas suffisamment, car ils sont loin de nous, ici sur le polygone, ou au Campus, loin de nous par leurs activités que nous ne comprenons pas toujours, loin de nous, par leur vocabulaire, qui ne nous est pas familier : Vous les chercheurs, les ingénieurs, et les techniciens. C'est également à vous que je m'adresse aujourd'hui, pour vous dire l'importance que j'accorde à votre mission, à l'ampleur de vos travaux, à la valeur de

vos découvertes. Pour souligner l'intérêt que j'attache à ce domaine, j'ai auprès de moi, au sein du Conseil Municipal, l'un des vôtres, Joël de Leiris, dont la mission est de créer des liens entre tous les partenaires, afin que soient mieux compris vos travaux, mieux résolus vos problèmes, mieux assimilés les nouveaux chercheurs français et étrangers qui demain seront les hôtes de notre ville et viendront renforcer ce secteur de la recherche et de la technologie dont nous sommes si fiers.

A ce titre, je voudrais évoquer ici le rôle économique que vous êtes appelés à jouer au cœur de notre cité. Certes, il y a loin de la recherche fondamentale, (qui est la mission de cet Institut), au développement technologique et industriel, mais, il doit y avoir, et nous ne le savons pas, et vous-mêmes ne le savez pas, il doit y avoir dans les études sur le magnétisme, les supraconducteurs ou la biologie et les nombreuses découvertes réalisées grâce aux neutrons, les produits industriels de demain.

En effet, le transfert des connaissances, le transfert des technologies est aussi une des spécialités de Grenoble : ce transfert du savoir passe d'abord par une meilleure connaissance des hommes et je soulignerai ici les travaux effectués à Grenoble dans le cadre du Club du Polygone, les rencontres entre chercheurs, décideurs et responsables locaux au sein du groupe de travail Grenoble-Technopole du Conseil Général de l'Isère.

Par toutes ces manifestations, par tous vos actes, vous êtes des éléments moteurs de l'Economie.

Tout d'abord, à titre de consommateur, il convient de souligner que chaque année l'Institut Laue-Langevin dépense environ 30 millions de Francs au plan régional. Ces dépenses sont également très importantes au C.E.N.G. et au C.N.R.S., et ceci ne pourra que s'amplifier avec la création du Laboratoire du Synchrotron : vous êtes également des producteurs. Certes, dans vos laboratoires, ce concept n'est pas une préoccupation quotidienne, néanmoins l'effort européen passe par le développement des technologies, elles naissent entre vos mains ; par votre activité quotidienne, vous produisez pour demain.

Vous tous qui êtes ici, depuis 1 an, 10 ans et même 20 ans, pour certains, vous remarquerez que comme à son habitude, Grenoble bouge, et bouge avec vous : le développement du polygone, les zones de transfert de technologie du C.E.N.G., de l'I.N.P.G, le Synchrotron, Europole, sont autant de projets auxquels la Ville s'intéresse et qu'elle soutient.

Nos efforts conjugués doivent nous permettre dans les mois et années à venir de construire une communauté, où les chercheurs, les ingénieurs, les industriels et les responsables locaux animés du même désir de rapprochement d'idées s'uniront pour bâtir l'Europe de la connaissance et de la Technologie.

Au moment où l'Institut fête son 20<sup>e</sup> Anniversaire, on peut affirmer, reprenant une publicité célèbre : Grenoble, c'est déjà demain.

## SPEECH BY H. MAIER-LEIBNITZ

J'aimerais tout d'abord exprimer mes sincères remerciements puisque j'ai la possibilité de parler au Jubilé de l'Institut Laue-Langevin. Cet organisme représente pour moi un très grand souvenir de ma vie.

Après la première conférence de Genève au sujet de l'utilisation pacifique de l'énergie atomique, la voie s'est ouverte pour beaucoup de pays pour recevoir ou construire des réacteurs nucléaires pour la recherche. Des relations amicales se

sont bientôt développées entre les chercheurs qui avaient déjà des réacteurs semblables aux Etats-Unis, au Canada, en Angleterre, en France et avec ceux qui se tournaient vers ce domaine pour la première fois. En



Prof. H. Maier-Leibnitz  
(Founder and first Director  
of the ILL : 1967-1972)

Europe Occidentale il y eut bientôt un groupe de travail des exploitants de réacteurs de recherche auquel participait également au tout début le Centre Nucléaire de la RDA à Rossendorf. Lorsque la construction d'un très grand réacteur a commencé à Brookhaven, supérieur à tous les autres réacteurs de recherche, on a réfléchi dans le cadre de l'OCDE (l'animateur étant M. Kowarski) pour savoir si un tel réacteur devait être construit en Europe. Les projets pour ceux-ci ont bien avancé, mais ont finalement échoué. L'idée de construire un tel réacteur maintenant "à la carte" avec une collaboration entre la France et l'Allemagne, au lieu d'avec tous les participants précédents, s'est d'abord soulevée en France où il faut citer les noms de Perrin, Hirsch, Horowitz et Goldschmidt. J'étais moi-même probablement le premier Allemand qui en avait entendu parler par le Chef du Cabinet de M. Perrin, M. Baïssas. Durant l'été de l'année 1964, une conférence a eu lieu de nouveau à Genève, comme la première, et là les Ministres Palewski et Lenz se sont rencontrés pour discuter du projet. A cette époque, la France pratiquait la politique de la chaise vide envers la Communauté Européenne. Tous étaient contents de pouvoir maintenant proposer quelque chose de positif.

Des discussions suivirent entre les scientifiques des deux pays et dans les Ministères compétents. Il en résulta une Convention Intergouvernement-

## The 20 years of the ILL - 1967-1987

tale par laquelle l'Institut Laue-Langevin fut créée pour une durée de quinze ans. Le CEA et le Gesellschaft für Kernforschung, maintenant le Kernforschungszentrum de Karlsruhe, s'en occupèrent. Un Comité de Direction composé de représentants des gouvernements, des deux centres ainsi que de scientifiques extérieurs, fut constitué pour les décisions de principe et pour les propositions financières.

Dès le début, à Grenoble, nous avons reçu beaucoup d'aide. Le Professeur Néel, qui a souvent joué le plus grand rôle dans l'aboutissement du projet, a mis à notre disposition les services du grand établissement qu'est le CENG, ainsi que des laboratoires du CNRS. Il faut mentionner M. Weil et M. Dautreppe, malheureusement décédés, M. Bertaut et M. Balligand, ainsi que le Préfet et le Recteur de l'Académie, M. Niveau. Les relations avec les autorités se sont bientôt traduites par des amitiés et d'excellentes relations sociales. L'amitié, c'est le mot clé pour le succès de notre entreprise.

Il était particulièrement important qu'un grand site très approprié ait été mis à notre disposition. On avait déjà pensé à l'époque qu'en cas de succès de l'Institut une extension pourrait s'avérer nécessaire, et je suis heureux que ce site soit suffisamment étendu pour permettre l'implantation d'un nouvel instrument et d'un Institut pour l'exploitation du rayonnement synchrotron.

L'Institut Laue-Langevin a démarré son exploitation avec l'arrivée des deux directeurs, M. Bernard Jacrot et moi-même, une secrétaire et, un peu plus tard, le Chef de l'Administration, M. Hasenclever. Tout d'abord, nous avons été logés dans un bâtiment du CENG où nous avons bénéficié de l'aide de l'Administration. L'existence du grand réacteur Siloé et de l'organisation associée ont été également très importantes pour nous.

Les études et la construction du réacteur lui-même ont été poursuivies comme projet à part, indépendamment du directeur de l'Institut, avec la collaboration décisive du CEA et de Karlsruhe. Dans ce contexte, M. Robert Dautray, M. Karl-Heinz Beckurts et M. Chatoux méritent d'être particulièrement mentionnés. Le projet du réacteur a été considérablement amélioré par rapport aux plans existants. La collaboration des ingénieurs des deux pays a fait ses preuves de façon extraordinaire à l'époque, ainsi que par la suite. Les souhaits des scientifiques pour les expériences envisagées ont été discutées avec le Groupe Projets, ce qui a permis d'aboutir aux meilleures solutions. Le rapport de Sécurité pour le grand réacteur qui a quand même été construit à proximité de la ville de Grenoble peut servir de modèle et a été pleinement corroboré par l'expérience de quinze années de fonctionnement.

J'aimerais en particulier signaler la collaboration constructive entre les membres du Comité de Direction. Les représentants gouvernementaux des deux pays ont fait confiance dès le début à l'expérience des deux organismes d'encadrement et ne sont pas intervenus dans ce domaine qui nécessite des connaissances spécialisées. Après une période courte de démarrage, une relation de confiance s'est établie entre le Comité de Direction et des directeurs de l'Institut. A l'époque, nous avons beaucoup appris des personnes déléguées à ce Comité, et nos propositions en ont été améliorées. Une atmosphère de confiance a été créée, ce qui a sûrement constitué une des bases les plus importantes pour le succès de l'Institut Laue-Langevin. De plus, le Comité avait une grande liberté pour ce qui concernait l'organisation. Selon le cas, des règlements parfois français, parfois allemands, ont été préférés, on a même pu introduire des procédures nouvelles.

Il faut considérer comme une circonstance heureuse que l'Institut Laue-Langevin ait eu, dès le début, la direction scientifique déjà en place et que, tout en poursuivant le développement du réacteur la préparation des expériences prévues a pu progresser avec tous les moyens nécessaires. Avec un investissement en personnes et en moyens qui dépassait celui d'Instituts comparables, tout a été mis en œuvre dès le début pour définir les expériences possibles et en général originales, et pour exécuter les travaux de développement en collaboration avec des scientifiques expérimentés d'autres Instituts dans le monde. Des relations étroites ont été nouées avec des laboratoires aux Etats-Unis (Brookhaven, Oak Ridge, Argonne, Los Alamos), avec Chalk River au Canada, avec des scientifiques britanniques, en particulier avec M. Egelstaff, avec la Suède et le Danemark, en France bien sûr avec Saclay et en Allemagne avec Karlsruhe, Munich et Jülich. Le développement des instruments en a beaucoup tiré profit. Quelques-uns ont même été construits en dehors

de Grenoble dans les Instituts déjà cités. Lors de la mise en marche, l'équipement expérimental a dépassé tout ce qui existait par ailleurs.

Dans ma mémoire cette époque a été caractérisée par une collaboration intense entre amis. Un effort particulier était nécessaire dans les domaines pour lesquels l'exploitation des neutrons paraissait prometteuse, mais à laquelle personne ne s'était encore adressé. Il n'est pas évident que quelqu'un qui a réalisé un travail important dans son domaine y introduise de nouvelles méthodes proposées par d'autres. Je me souviens avec gratitude de nombreuses discussions qui ont contribué à surmonter cette difficulté.

Probablement d'une importance décisive pour la réussite a été la décision que nous avons prise dès le début et qui a été soutenue par le Comité de Direction : l'Institut ne devait pas être une Institution autonome scientifiquement, dans laquelle des spécialistes de tous les domaines utiliseraient des neutrons de manière indépendante. Il devait avoir une fonction de service, tout en gardant l'autonomie des scientifiques travaillant à Grenoble. Tous les chercheurs des deux pays ainsi que beaucoup de citoyens d'autres pays, dont le travail était important pour le succès de l'entreprise à Grenoble, devraient bénéficier de toutes les facilités et de toute assistance afin d'utiliser les neutrons du grand réacteur pour leur travail. Aucun frais ne devrait leur être facturé, oui, l'Institut était prêt à verser également des frais de missions dans les cas où les organismes d'origine n'auraient pas pu facilement le faire. La grande majorité du personnel scientifique de l'Institut a reçu des contrats de cinq ans afin d'assurer un échange avec d'autres Instituts. Des postes à durée non limitée ont été accordés à ceux dont la présence à Grenoble avait une importance particulière pour le travail des autres, ou qui pouvaient donner une aide indispensable aux groupes de visiteurs en s'occupant des grands instruments. Un tel système rencontre facilement des résistances. Je suis particulièrement content de pouvoir dire qu'il a pu être mis en place et a fait ses preuves.

Tout le monde est sûrement très satisfait aujourd'hui du choix de Grenoble comme site d'emplacement de l'Institut Laue-Langevin. J'aimerais dire cela en particulier pour les chercheurs et techniciens allemands pour lesquels la perte de leur environnement familial a été compensée par la beauté de leur nouveau lieu de travail, par l'intérêt d'un travail qui en vaut la peine et par les contacts et les amitiés avec leurs collègues français et avec des visiteurs d'autres pays.

Je crois que tous ceux qui travaillent à l'Institut à Grenoble ont développé un sentiment de solidarité avec leurs collègues en Europe, et je crois qu'un modèle est né ici pour l'amitié franco-allemande, qui a une telle importance pour la réussite de l'Europe. Nous nous sentons tous Européens. Il est vrai que nous avons commencé par la collaboration entre deux pays, puisque cela est plus facile à organiser. La procédure "à la carte" a des avantages indéniables, si on n'oublie pas la possibilité d'un élargissement éventuel. Entre temps, l'Institut a eu trois partenaires et entretient une collaboration avec de nombreux membres d'autres pays. Le nouvel Institut à Rayonnement Synchrotron aura davantage de participants et je crois que je ne vais rencontrer aucune contradiction si je dis que les scientifiques, par leur nature, sont une espèce de citoyens du monde, mais qu'une collaboration européenne est reconnue souhaitable et soutenue par nous tous, chacun sachant qu'en cela il sert en même temps son propre pays.

En rétrospective, j'ai le sentiment d'une grande gratitude d'avoir pu collaborer dans cette entreprise et d'avoir pu peut-être faire quelque chose pour la coopération entre nos pays. Mais avant tout, je dois être reconnaissant envers tout ceux qui ont aidé dans cette entreprise et qui ont donc accompli la réussite visible d'aujourd'hui.



Dr. J. Horowitz (Former Director of the Institute of Fundamental Research, CEA)

## SPEECH BY J. HOROWITZ

L'ILL : un modèle d'organisme multinational de recherche fondamentale.

En ce vingtième anniversaire de la fondation de l'ILL il est naturel d'évoquer avant tout les succès techniques et scientifiques de cette entreprise. Succès technique d'abord car la remarquable réussite de la réalisation et de l'exploitation du Réacteur à Haut-Flux et des systèmes associés a constitué un préalable au remarquable succès scientifique qui a fait de l'ILL le leader mondial incontesté dans le domaine des recherches avec les faisceaux de neutrons.

Malgré la prééminence de ces aspects scientifiques et techniques, il semble opportun et instructif d'évoquer en ce vingtième anniversaire les problèmes de structure, de statut et d'organisation qui ont reçu à l'ILL des solutions à beaucoup d'égards exemplaires.

La plupart des organismes de recherche multinationaux connaissent actuellement en Europe des difficultés liées en grande partie à des questions de personnel, difficultés que le statut de l'ILL a permis d'éviter.

L'ILL devait être implanté au voisinage immédiat de plusieurs établissements de recherche français ayant des activités analogues aux siennes. De plus, sa vocation de laboratoire de service devait y amener de nombreux expérimentateurs extérieurs. Les fondateurs de l'ILL ont estimé essentiel d'éviter des disparités injustifiées entre la situation des agents de l'ILL et celles de leurs collègues grenoblois ou extérieurs. Pour cette raison, et aussi dans un souci général d'économie, il a été décidé de créer l'ILL comme une société civile de droit français et de ne pas lui donner l'un de ces statuts internationaux dont les inconvénients pour des organismes de recherche, manifestes aujourd'hui, étaient déjà prévisibles il y a vingt ans.

Il eût été déraisonnable de vouloir élaborer un statut du personnel ou une convention de travail qui génère pour un Institut de moins de 500 agents et d'obliger sa direction à négocier périodiquement avec des partenaires sociaux des règlements et des accords salariaux particuliers.

Aussi décida-t-on d'adopter pour l'ensemble de ces questions les règles en vigueur au Commissariat à l'Energie Atomique, mais à part quelques points spécifiques comme les indemnités d'expatriation, la durée limitée des contrats de la majorité des physiciens et du mandat des directeurs, etc. Dans l'ensemble ces dispositions se sont révélées efficaces et satisfaisantes sans toutefois pouvoir remédier à tous les inconvénients du manque de mobilité qui s'est progressivement généralisé en Europe.

Dès que l'accord de principe franco-allemand pour la création de l'ILL fut acquis, le travail a effectivement débuté pour aborder les phases de projet et de construction et pour commencer à mettre en place les structures. Aussi au moment de la signature des textes légaux, il y a vingt ans, beaucoup avait déjà accompli aux plans scientifique et technique et bien des dispositions de l'organisation étaient déjà, de facto, entrées en vigueur. Ce développement rapide de l'entreprise n'avait été rendu possible que par la collaboration confiante et efficace qui s'est établie dès l'abord entre les différentes parties concernées, les ministères de la recherche des deux pays ainsi que le Kernforschungszentrum

Karlsruhe, le Centre National de la Recherche Scientifique et le Commissariat à l'Energie Atomique, futurs Associés dans la Société ILL, chacun œuvrant selon ses compétences et ses responsabilités.

Il est certain que le caractère seulement binational a été un facteur de simplification lors de la création de l'ILL. Mais il convient de souligner que dès que le principe de l'adhésion de la Grande-Bretagne à l'ILL fut décidé, tous les problèmes concrets ont été réglés en moins de quatre mois de sorte que le Science Research Council devint un Associé effectif au début de 1973, environ un an et demi avant la signature des accords gouvernementaux.

Au plan scientifique chacun des trois pays membres apporta à l'ILL une contribution spécifique ce qui réalisa une complémentarité très bénéfique. Dans une certaine mesure, il en fut de même pour la direction et la gestion de l'Institut.

La qualité et la sagesse des statuts ne pouvaient à elles seules garantir ni le bon fonctionnement de l'Institut ni des relations confiantes et constructives entre la direction de l'ILL et les Associés dans le cadre du comité de direction et de ses différents sous-comités. Un élément éminemment favorable était que les Associés avaient par vocation le double souci de veiller à une stricte économie de moyens et d'assurer à leur communauté scientifique les meilleures possibilités expérimentales. De plus, les Associés ont fait l'effort continu de s'intéresser au détail des problèmes scientifiques, techniques et administratifs de l'ILL, évitant ainsi l'exercice d'une tutelle de type technocratique. L'un des exemples de la sollicitude des Associés pour le maintien et le développement du potentiel scientifique et technique de l'ILL a été sans doute le programme de modernisation dont ils ont pris l'initiative et dont ils ont obtenu le financement de leurs gouvernements en plus des budgets ordinaires malgré un contexte économique parfois difficile.

Le programme de modernisation se termine maintenant et, comme l'exige la crédibilité de cette politique, les budgets annuels ainsi que les effectifs vont retrouver leur niveau ordinaire en attendant éventuellement un nouvel effort exceptionnel qui pourrait s'imposer dans quelques années.

Pour un organisme multinational de recherche fondamentale, le fait de n'avoir que deux ou trois, à la rigueur quatre pays membres, apportant des contributions financières égales et ayant des intérêts scientifiques comparables, offre incontestablement des conditions très favorables pour la gestion de l'ensemble des problèmes scientifiques, administratifs et financiers. Il est en particulier normal, dans ce cas, que l'accès aux instruments ne soit déterminé que par des critères scientifiques et pour les décisions administratives et budgétaires la règle de l'unanimité présente alors peu d'inconvénients.

Certes, l'ILL peut accepter des projets d'expériences émanant de pays tiers mais présentant un intérêt scientifique spécial. Néanmoins, depuis plusieurs années, se pose la question de l'ouverture de l'ILL à des pays dans lesquels le volume des besoins en matière de diffraction neutronique est sensiblement inférieur à celui des trois pays membres.

L'accord qui vient d'être conclu avec l'Espagne apporte une solution très satisfaisante à ce problème puisqu'en échange d'une contribution faible, ce pays obtient un accès aux installations de l'ILL ainsi qu'à ces instances scientifiques sans être impliqué dans la gestion administrative et technique de l'Institut.

L'ILL a vingt ans. Le programme de modernisation vient de lui donner une nouvelle jeunesse. Du point de vue scientifique et technique rien ne s'oppose à ce qu'il conserve pendant vingt ans encore sa place de leader mondial dans le domaine de la diffraction neutronique et à ce qu'il s'ouvre plus largement à d'autres pays. Mais pour que le rapport efficacité/coût se maintienne à un niveau sans doute exemplaire, il importe que son organisation et ses structures conservent leurs qualités actuelles.



Prof. E.W.J. Mitchell (Chairman of the Science & Engineering Research Council, U.K.)

## SPEECH BY E.W.J. MITCHELL

It is a particular honour and pleasure for me on behalf of SERC and especially personally to be here today, because of the occasion we are celebrating. The UK was conspicuous by its absence - that of course reflected no lack of interest from UK scientists in the tool of neutron scattering, but represented its decisions about the way to provide neutron scattering facilities by successive UK Governments between 1959 and 1973. Finally in 1973, the UK became a de facto partner as a result, incidentally, of a decision by the present Prime Minister, who was at that time Secretary of State for

I know that I can speak on behalf of all UK scientists involved at that time, but we will always be grateful for the tolerance that our French and German colleagues showed in that period of construction between 1966 and 1972; and especially that shown by the founding Director, Professor Heinz Maier-Leibnitz.

I take my presence today as a further example of that tolerance because there is a real sense in which the celebration is yours - French and Germans - commemorating the foresight and resolve of that agreement in 1967. The only credibility we can have to be here today - and I hesitate to make any light-hearted remarks in such a formal proceeding - is that we did pay our share of the historic capital and that gave us a sort of retrospective membership, and being here today at least allows me to praise the French and Germans and the action in 1966, so that you do not have to confine the occasion to mutual self-adulation, which incidentally you have every right to do. The UK membership was agreed in 1973 and for several months in a very informal and effective way I acted as an unpaid Associate Director, as Professor Mössbauer will recall, with incidentally surprising powers bestowed on me by the, as it was then SRC. Indeed, sometimes I think I was given more powers in those few months that I possess now as the Chairman of the SERC. But during that time we slotted in UK proposals and we interviewed staff and did various things until Dr. Lomer, the first British Associate Director took office in the Spring or thereabouts of 1974.

Education and Science, and with the formal UK signature in 1974, by Mr Prentice.

## The 20 years of the ILL - 1967-1987

The discussions as has been said about a European High Flux Beam Reactor started under the European Nuclear Energy Agency (E.N.E.A.) part of the O.E.C.D. in 1959 or thereabouts. There was general enthusiasm and interest and I remember a particular meeting at Harwell in 1960, at which both Cockcroft and Covosky played leading roles. The UK stalwarts of the time were Professor Anderson, a chemist, Bacon Egelstaff, Lomer, Mitchell and Squires, and we wrote a report about High Flux Beam Reactors to our funding bodies in 1963. I think it was really Professor Anderson who was responsible in the UK for interesting the chemists who later became very well known in the field, Professors Allen, Fender, White and the late Tom Waddington.

SRC was active in renting Harwell facilities from 1966 and we were operating an allocation scheme to a growing community from that time. Many of us indeed in the UK, those from the Universities, learned the beginnings of techniques as summer visitors at atomic energy establishments at Harwell or Chalk River during the summers, and it was certainly recognised by those involved from the Universities at that time that neutron scattering represented a general tool of science to be used where other techniques will not work. It reminds one of the Heineken's beer advertisement on British television, which reaches the parts of the body that other beers do not reach, and so neutrons have this ability to solve problems which other radiations will not solve. It was clear however, and this is a serious point which certainly Professor Maier-Leibnitz and the French colleagues also at that time were aware of, that it was not a special atomic energy matter - the technique was a general scientific importance.

The encouragement in the UK, as far as its community was concerned, informally at first and then subsequently with the agreement of the then SERC, gradually built a large community, and of course because of the scientific importance that one has referred to, this community was eventually going to produce its own demands independent of atomic energy institutes and the delay between 1963, when as I said there was a definitive recommendation, and 1973 was partly the Government's trying to assimilate the ubiquitous importance of neutron scattering and deal administratively with it, and indeed I think also at the beginning of a questioning of what the role of atomic energy laboratories actually was in the longer term. Nevertheless, the development of the community went on apace, with a major interest developed by the UK chemists, as well as the physicists involved in solid state and materials, and in this period, 1973 and 1974, we came to the Institute both grateful for your tolerance, which I have already referred to, but absolutely excited by the superbly engineered reactor and the brilliantly conceived instruments.

I think that for our community it was the best thing that could have happened. Using the medium flux facilities, we had set out to use neutrons in those earlier years and we think we had helped to show that the predictions in all the reports of the early '60s were right: that neutrons were invaluable in chemistry and certainly physics, and even then there were interests developing in biology. But in doing that, and I think that French and German colleagues would agree that it was moderately successful, our instrument thinking and development relatively speaking had taken a back seat and our eyes were opened by coming here and observing the concepts developed by Professor Maier-Leibnitz coming to fruition in the wonderful new instruments in 1974. So at last we got to the present situation with the 3 associates and now of course the added membership association of Spain.

The French and Germans with whom we discussed at the time or, at least before the agreement, asked whether we would want to change the name of the Institute, and UK officials said shouldn't the name be changed, but the scientists involved unhesitatingly said that the name should not be changed, and that the decisive act of France and Germany in starting this Institute should be permanently recognised by the title, Institut Laue-Langevin.

One of those random events of life I think that one can bring out on this sort of occasion, as an aside - I did my PhD in Bristol and of course Professor Enderby, Associate Director, is the Head of the Department at Bristol, on leave, and the H.H. Wills Physics Laboratory at which we at different times then worked was opened in 1925, and on the walls of the old coffee room in the laboratory is a picture of the opening at which Langevin was present, which I think is interesting, and it was a nice little return gesture when the Institute decided to call this room the James Chadwick Amphitheatre.

The collaboration has, I think, been a great success for all the reasons which Professor Maier-Leibnitz and Dr. Horowitz have referred to. Of course, circumstances have changed, the flow out into Universities has been more difficult in recent years, I think in all 3 countries, but the Institute has adapted itself and remains an exciting and young body. The older hands may occasionally be frustrated by the user institute concept but let me say that it is your life blood, the continual involvement of young scientists coming through the Institute. Some of you will know that the SERC Council met her last May. I brought them to show them the ILL and on the return journey we made a visit to CERN. At a subsequent discussion of these visits afterwards, Council Members, irrespective of discipline, were enormously impressed by the liveliness and excitement here in comparison with what they saw at CERN, which by its nature of course has very long lead times and a myriad of separate developments, which are associated with big science. At such a formal occasion perhaps I should not say any more.

The scientific record here speaks for itself in all fields. The users and the scientific staff have made advances leading the world in the study of biological systems, membranes and structures in amorphous systems, liquids, molten salts, metallic glasses in excitations, magnetic and vibrational structures, in polymers, in materials problems and in neutron related nuclear physics.

The Institute has also enabled advances to occur at the same time in pursuing fundamental studies of neutron characteristics and of special programmes requiring extended periods. As a user, or at least as a former user, a significant part of nearly 15 years of my scientific life has come from an interaction with this Institute. So, I think that I am in a good position to say that the success we have all spoken of comes not only from the Directors but also from the dedication of all the staff in the Institute and especially, if I may say so, the Senior and Staff Scientists with whom it has been a pleasure to be associated and work. They have set a pattern of unselfish interactions that has been crucial to the success, and which other people have followed.

What of the future? The 1974 agreement said that the next High Flux Source in Europe should be in the UK. Now it is built and we see it as very complementary and we look forward to welcoming you to it, and I think then confirming the continuing European leadership in the application of neutrons to science and enabling these very high flux facilities and activities associated with them to be opened to an even wider European membership. That is for the future. On this occasion then let me thank you ILL and may your success continue and extend.

## The 20 years of the ILL - 1967-1987



*The signature of the intergovernmental agreement on 19 January 1967 by the ministers G. Stoltenberg and A. Peyrefitte.*



*Monsieur J. Mingasson (left), Préfet de l'Isère and Prof. W. Gläser, ILL Director*



*The ceremony in the Chadwick amphitheatre of the ILL*

# The 20 years of the ILL - 1967-1987

Following the ceremony a drink was offered to the dignitaries



From left to right: M. Suscillon (Head of the CEN-Grenoble); B. Dreyfus (CEN-Grenoble) and J. Winter (Head of Fundamental Research at the CEN-Grenoble)



R. Mössbauer (right) (TU-Munich) talking to A. Freund (ILL)



T. Springer (left) (KFA-Jülich) and E.W.J. Mitchell



H. Maier-Leibnitz (left) and R. Dautray (first Head of the High Flux Reactor Project)



The second neutron guide hall was an ideal place to offer a drink to all ILL staff



R. Mössbauer (right) had an animated discussion with T. von Egidy (centre) and W. Mampe



B. Standke presenting H. Maier-Leibnitz (founder of the ILL) with the reward "Grand Maître de la Confrérie du Taste Neutrons". On this occasion she read an excerpt of "De bello neutronibus" by Julius Jacquiminius: "Dès son entrée dans la vie scientifique, le proconsul Heinz Maier-Leibnitz manifesta le plus grand dynamisme dans la lutte pour la connaissance du neutron. Après des débuts prometteurs en Germanie, il eut en 1967 la géniale idée d'attirer ces particules dans un vaste polygone désert situé au confluent du Drac et de l'Isère, afin de les pousser à s'y reproduire. Franchisant les frontières à la tête d'une légion de jeunes physiciens parmi lesquels bon nombre de Bavarois plus ou moins disciplinés, il rejoignit à GRATIANOPOLIS quelques peuplades scientifiques gauloises encore moins disciplinées, dont certaines avaient même consenti à quitter Lutèce pour venir s'exiler dans les neiges delphinales. C'est là qu'il assista avec étonnement et intérêt aux grandes vèpres estudiantines de Mai 1968. Enfin, prêt au combat, et déjà assuré du renfort des légions anglo-saxonnes, il passa à l'attaque dès 1971, traquant sans répit ces malheureux neutrons et s'en servant même comme projectiles. Adaptant à cette lutte son expérience culinaire bien connue, il les fit rôtir en source chaude, congeler en source froide, s'écouler en collimateurs, écraser en hyperbars, presser en champs magnétiques et antineutrons des combats fratricides et anihilateurs. Ayant dû supporter pendant toute cette campagne, des administrateurs avarés, des techniciens farfebus, un Comité Directeur parfois sourcilieux, il entra en Germanie le front ceint des lauriers de la victoire. Il restera pour toujours dans le souvenir de ceux qui ont eu la chance de vivre et de travailler avec lui. En conséquence, nous vous INTRONISONS GRAND MAITRE DE LA CONFRERIE DU TASTE NEUTRONS."



W. Gläser trying hard to blow out the candles surrounding the anniversary cake



An attractive ballet presented by B. Standke and her "show-girls" marked the event.

## Visits and events in 1987

*The following personalities visited the ILL in 1987*



*Mr. Song Jian, President of the Chinese Commission for Science and Technology, visited the ILL on 9 April 1987. He was accompanied by W. Gläser during his tour through the experimental halls of the ILL*

*K.H. Narjes (centre), Vice-President of the Commission of the European Community, visited the ILL on 18 May 1987. On his tour through the Institut he was accompanied amongst others by J. Mingasson (front), Préfet de l'Isère*



# Bericht des Direktors

**W**ährend ich diesen Bericht über 1987 schreibe, sind die Vorbereitungen für unser Programm in 1988 schon in vollem Gange. Das vergangene Jahr war für das ILL ein besonderes und ereignisreiches Jahr. Mein Bericht kann nur fragmentarisch sein.

Am 19. Januar konnten wir das 20-jährige Jubiläum der Gründung des Instituts feiern. Die Teilnahme zahlreicher Persönlichkeiten aus Politik und Wissenschaft an dieser Veranstaltung und ihre Würdigung des Erreichten erfüllt uns mit Stolz. Unsere Aufgabe wird es sein, die führende Position des ILL auf dem Gebiet der Neutronenforschung zu erhalten und weiter auszubauen.

Die einzigartigen Forschungsmöglichkeiten haben zu einem grossen Interesse der Wissenschaftlergemeinden weiterer Länder an einer Beteiligung am ILL geführt. Anfang 1987 ist Spanien wissenschaftliches Mitglied des ILL geworden, und Verhandlungen über eine wissenschaftliche Mitgliedschaft der Schweiz sind weitgehend abgeschlossen.

Zentrale Einrichtung des Instituts ist der Hochflussreaktor, der nach dem Modernisierungsprogramm zuverlässiger denn je läuft. Ende 1987 konnte die neue horizontale kalte Quelle wie vorgesehen eingebaut und getestet werden. Für diese ausserordentliche Leistung gebührt den beteiligten Technikern und Ingenieuren besondere Anerkennung.

Planung und Bau der Instrumente für die zweite kalte Quelle konnten weiter vorangetrieben werden. Der Aufbau des 3-Achsenpektrometers IN14 ist fast abgeschlossen, ebenso ist der Aufbau des (n,n̄) Experiments, eine Kollaboration der Universitäten Padua, Pavia, Heidelberg und des ILL, weitgehend fertiggestellt. Die Projekte IN15, ein hochauflösendes Spin-echospektrometer und IN10C, ein intensitätsstarkes Rückstreu-spektrometer befinden sich in einem fortgeschrittenen Planungsstadium. Weiter wurde beschlossen, ein neues Kleinwinkelstreuinstrument D22 zu bauen. Obwohl sich damit 4 neue Instrumente in der Planung bzw. im Bau befinden, erfordert das Aufkommen neuer Anwendungsmöglichkeiten der Neutronenstreuung, wie z.B. der Anwendung der Totalreflexion zur Untersuchung von Oberflächenschichten oder der Brillouin-Spektroskopie zur Untersuchung der Dynamik von ungeordneten Systemen weitere Anstrengungen bei der Methodenentwicklung. Nicht zuletzt stellen dabei die Erfahrungen aus S-Experimenten eine wertvolle Hilfe dar.

Schliesslich wird die Entscheidung zur Erneuerung der zentralen Rechenanlage zu einer wesentlichen Verbesserung der für alle Experimente wichtigen Datenerfassung und -verarbeitung führen.

Trotz der Einschränkungen durch die Projektarbeiten für die zweite kalte Quelle konnten im Jahr 1987 5 Reaktorzyklen planmässig durchgeführt werden. Von den insgesamt eingereichten 1056 Experimentivorschlägen mit einer gewünschten Strahlzeit von 10497 Tagen konnten 742 Experimente mit einer Gesamtstrahlzeit von 5347 Tagen verwirklicht werden.

## Wissenschaftliches Programm und Ergebnisse

Auf die 1987 durchgeführten wissenschaftlichen Arbeiten und die erzielten Ergebnisse wird in den anschliessenden Berichten der Colleges näher eingegangen. Hier seien nur einige besonders interessante Ergebnisse summarisch erwähnt.

Nach der Entdeckung der neuen Hochtemperatursupraleiter durch Bednorz und Müller war klar, dass Neutronenstreuung zur Aufklärung ihrer mikroskopischen Eigenschaften nützlich sein kann. Das Institut hat deshalb für diesen Problemkreis ausserhalb der Reihe kurzfristig Messzeit zur Verfügung gestellt, insgesamt etwa 100 Strahltag auf verschiedenen Instrumenten. Auf diese Weise konnte das ILL einige wichtige Beiträge leisten; so konnte die Struktur des "100 K" Supraleiters  $\text{YBa}_2\text{Cu}_3\text{O}_7$  und insbesondere die Anordnung der Sauerstoffdefekte erstmalig bestimmt werden. Streuung von polarisierten Neutronen an dieser Verbindung und Polarisationsanalyse führten zu dem Ergebnis, dass die gesamte magnetische Streuung verschwindend klein ist und folglich magnetische Kopplungsmechanismen unwahrscheinlich sind. Dagegen wurde z.B. in  $\text{GdBa}_2\text{Cu}_3\text{O}_7$  magnetische Ordnung bei tiefen Temperaturen gefunden. Schliesslich liegen inzwischen auch die Ergebnisse einer ganzen Reihe von gitterdynamischen Untersuchungen an  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$  vor.

Ausser in diesem adhoc-Programm wurde im normalen Messprogramm eine beachtliche Zahl schöner Ergebnisse erzielt, z.B.

- über hochenergetische magnetische Anregungen in 3d-Übergangsmetallen (heisse Quelle) und über tiefliegende Anregungen in antiferromagnetischen Garnets (kalte Quelle)
- bei den Strukturuntersuchungen über die quasikristalline Struktur von Al-Li-Cu-Legierungen und über die Struktur von Heliumhydraten
- bei den ungeordneten Systemen über die Dynamik des Glasübergangs und über das Anregungsspektrum von  $^3\text{He}$  -  $^4\text{He}$ -Mischungen
- bei den biologischen Systemen mit Hilfe der Kontrastvariationsmethode über das Verhalten der RNA Polymerase bei der Transkription
- aus dem Bereich der Chemie sind die Oberflächenreflexionsmessungen an Polymerlösungen und der erstmalige Nachweis von Formrelaxationsmoden kleiner Tröpfchen in Mikroemulsionen zu nennen
- bei den Arbeiten im Bereich der Kern- und Teilchenphysik müssen insbesondere die Entdeckung neuer sehr neutronenreicher Spaltprodukte wie z.B.  $^{76}\text{Ni}$  (LOHENGRIN) und die ersten Bestimmungen extrem kurzer Lebensdauern mit dem Kristallspektrometer GAMS4 erwähnt werden.

## Personalia

Im vergangenen Jahr hat es im Institut im allgemeinen keine grossen personellen Veränderungen gegeben. Zu erwähnen ist, dass die Leitung der Abteilung EDEX von C. Janot an A. Heidemann übergegangen ist.

Auch 1987 sind Mitarbeiter des Instituts ausgezeichnet worden oder haben Rufe erhalten. D. Richter ist mit dem Walter Schottky-Preis für Festkörperphysik der Deutschen Physikalischen Gesellschaft ausgezeichnet worden. W. Stirling hat einen Ruf als ordentlicher Professor an die Universität Keele erhalten und angenommen. O. Schärpf wurde mit der Wahrnehmung einer Lehrstuhlvertretung an der Technischen Universität München beauftragt. Wir betrachten diese Anerkennungen auch als Auszeichnung des ganzen Instituts.

Im Berichtszeitraum haben wieder eine Reihe bekannter Politiker und Wissenschaftler das Institut besucht. Neben den Teilnehmern an der 20-Jahrfeier sind u. a. zu nennen der Besuch einer chinesischen Regierungsdelegation unter Leitung des chinesischen Forschungsministers Song Gian sowie der Besuch des Vizepräsidenten der Kommission der Europäischen Gemeinschaft K.-H. Narjes.

## Zusammenarbeit mit der ESRF

Mit der Entscheidung, die Europäische Synchrotron-Strahlungsquelle in unmittelbarer Nachbarschaft des ILL anzusiedeln, wurden die Weichen für die Schaffung eines einzigartigen Zentrums für die Erforschung der kondensierten Materie gestellt. In der seit 1986 laufenden Vorbereitungsphase der ESRF hat das ILL auch 1987 das Mandat zur Vertretung der ESRF bis zu deren rechtsförmlichen Gründung wahrgenommen und die Gründungsmannschaft in vielerlei Hinsicht mit seinen Einrichtungen und Diensten unterstützt. 1987 wurden bei der Vorbereitung der zukünftigen Zusammenarbeit auf der Basis gegenseitiger Freiwilligkeit beachtliche Fortschritte erzielt. So wurde vor allem Einigung über die Schaffung eines gemeinsamen integrierten Forschungsgeländes erzielt, das Voraussetzung für eine effektive und ökonomische Zusammenarbeit ist und das durch einen neuen gemeinsamen Pachtvertrag mit dem CEA begründet werden soll.

Es sei angemerkt, dass das ILL inzwischen einen Teil des in die künftige ESRF-Zone fallenden Geländes freigemacht hat und dass die Finanzierung der Räumungsarbeiten des übrigen Geländes inzwischen gesichert ist.

Darüberhinaus wurde eine Reihe von gemeinsamen Arbeitsgruppen gegründet, die die konkreten Möglichkeiten der künftigen wissenschaftlichen, technischen und administrativen Zusammenarbeit im einzelnen prüfen sollen und deren Arbeit auch schon zu einigen attraktiven Vorschlägen geführt hat, die bei der Bau- und Finanzplanung der ESRF berücksichtigt werden sollen. Es bleibt zu wünschen, dass die ESRF möglichst bald durch Unterzeichnung der entsprechenden Regierungsabkommen einen legalen Status erhält.

*Ich habe die traurige Pflicht, vom Tod unseres ehemaligen Mitarbeiters Mr. Stuart Wilson zu berichten. Mr. Wilson war über viele Jahre als Verantwortlicher des Diffraktometers D16 ein aktives Mitglied der Kristallographiegruppe und war im Frühjahr dieses Jahres in den verdienten Ruhestand getreten. Wir werden ihn in besonderer Erinnerung behalten.*

W. Gläser

# Director's Report

**A**s I write this report on 1987, the preparations for our 1988 programme are well advanced. 1987 was a particularly eventful year for the ILL. My report can only be fragmentary.

On 19 January we celebrated the 20th Anniversary of the foundation of the Institut Laue-Langevin. The participation of numerous personalities in politics and science on this occasion and their praise for what has been achieved is a source of pride for us. It is our aim to maintain and enhance the ILL's leading position in the field of neutron research.

The unique research facilities have resulted in considerable interest among the scientific communities of other countries in joining ILL. At the beginning of 1987 Spain took up scientific membership of the ILL, and negotiations on Swiss scientific membership are almost concluded.

The ILL's central facility is the High Flux Reactor, which is operating with an even higher degree of reliability since the Modernisation Programme. At the end of 1987 the new Horizontal Cold Source was installed and tested as planned. The technicians and engineers concerned deserve particular recognition for this excellent achievement.

Further progress has been made with the planning and construction of the instruments for the Second Cold Source. The installation of the three-axis spectrometer IN14 is almost completed, as is that of the  $n, \bar{n}$  experiment, a collaboration between the universities of Padua, Pavia and Heidelberg and the ILL. The projects IN15, a high resolution spin-echo spectrometer, and IN10C, a high intensity backscattering spectrometer, are at an advanced planning stage. It has also been decided to build a new small angle scattering instrument D22. Although this means that there are four new instruments at the planning or construction stage, new applications of neutron scattering, such as the application of total reflection to the study of surface layers or the use of Brillouin spectroscopy to study the dynamics of disordered systems, necessitate further efforts in the development of methods. The experience gained with "S" experiments is also a valuable help in this.

Finally the decision on the renewal of the central computer will result in a considerable improvement in data acquisition and treatment, which is important for all experiments.

Despite the limitations due to the preparatory work for the Second Cold Source, five reactor cycles were completed in 1987 according to plan. Of the total of 1056 experiment proposals requesting beam time of 10497 days, it was possible to carry out 742 experiments with a total beam time of 5347 days.

## Scientific programme and results

Details of the scientific work carried out in 1987 and of the results obtained are given in the reports from the colleges which follow. Here only a few particularly interesting results are summarized.

After the discovery of the new high temperature superconductors by Bednorz and Müller, it was clear that neutron scattering can be useful for clarification of their microscopic properties. The ILL exceptionally made available beam time at short notice for these problems, a total of approximately 100 days on various instruments. In this way the ILL was able to make some important contributions; for example it was possible to determine for the first time the structure of the "100 K" superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_7$ , and in particular the arrangement of the oxygen defects. Polarised neutron scattering on this compound and polarisation analysis showed that

the total magnetic scattering is extremely small, so that magnetic coupling mechanisms are unlikely. On the other hand in  $\text{GdBa}_2\text{Cu}_3\text{O}_7$ , for example, magnetic ordering was found at low temperatures. Finally results have now been obtained for a number of lattice dynamic investigations of  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ .

In addition to this ad hoc programme a considerable number of excellent results were obtained in the normal measuring programme, e.g.

- on high-energy magnetic excitations in 3d transition metals (hot source) and on low-energy excitations in antiferromagnetic garnets (cold source)
- in studies of the quasi-crystalline structure of Al-Li-Cu alloys and on the structure of helium hydrates
- in disordered systems on the dynamics of the glass transition and the excitation spectrum of mixtures of  $^3\text{He}$  and  $^4\text{He}$
- in biological systems with the aid of the contrast variation method on the behaviour of the RNA polymerase on transcription
- in the chemistry field, surface reflection measurements on polymer solutions and the first evidence of form relaxation modes of small droplets in microemulsions
- in the work in the field of nuclear and particle physics, particular mention should be made of the discovery of new, very neutron-rich fission products such as  $^{76}\text{Ni}$  (LOHENGRIN) and of the first determinations of extremely short lifetimes with the crystal spectrometer GAMS4.

## Personnel

In general there were no major staff changes at ILL during the year. The replacement of C. Janot by A. Heidemann as head of the Instrument Operation Department should be mentioned.

Again in 1987 members of the ILL staff received honours or prizes. D. Richter was awarded the Walter Schottky prize for Solid State Physics by the Deutsche Physikalische Gesellschaft. W. Stirling accepted a chair at the University of Keele. O. Schärpf was requested to occupy a chair at Munich Technical University on a temporary basis. We consider these distinctions as an honour for the entire Institut.

During the report period a number of well-known political and scientific personalities have again visited the ILL. In addition to those who attended the 20th Anniversary celebrations, mention should be made of a Chinese government delegation led by the Chinese Minister for Research Song Gian, and a visit of the Vice-Chairman of the Commission of the European Community K.-H. Narjes.

## Collaboration with the ESRF

The decision to establish the European Synchrotron Radiation Facility in the immediate vicinity of the ILL provided the conditions for the creation of a unique centre for research on condensed matter.

During the preparatory phase of the ESRF, which started in 1986, the ILL continued in 1987 to act on behalf of the ESRF until its formal foundation, and supported the project team in many ways with its installations and services. Considerable progress was made in 1987 in the preparation of the future collaboration on a voluntary basis on both sides. In particular, agreement was reached on the establishment of an integrated common research site, which is a precondition for effective and economic cooperation, and which is to be based on a new joint lease with the CEA.

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It should be mentioned that the ILL has now released part of the site for the future ESRF zone, and that the finance is now ensured for the clearance work on the remainder of the site.

A number of joint working groups were also set up to examine in detail the practical possibilities for future scientific, technical and administrative cooperation. Their work has already led to a number of attractive proposals, to be taken into account in the ESRF's construction plans and financial planning. It is hoped that the ESRF will achieve legal status as soon as possible with the signature of an intergovernmental agreement.

*I have the sad task of reporting the death of our former colleague Stuart Wilson. For many years, Mr. Wilson was responsible for the diffractometer D16 and was an active member of the crystallography group. In Spring 1987 he took a well-earned retirement. We shall keep a particular remembrance of him.*

*W. Gläser*

# Rapport du Directeur

**T**andis que j'écris ce rapport sur l'année 1987, les préparatifs pour notre programme de 1988 sont déjà bien avancés. L'année dernière a été pour l'ILL une année particulièrement riche en événements. Mon rapport ne peut en mentionner qu'une partie.

Le 19 janvier nous avons pu fêter le 20<sup>e</sup> anniversaire de la création de l'Institut Laue-Langevin. La participation à cet événement de nombreuses personnalités du monde politique et scientifique et leur reconnaissance du travail accompli nous remplissent de fierté. Notre tâche sera de maintenir et d'améliorer la position de pointe de l'ILL dans le domaine de la recherche neutronique.

Les possibilités exceptionnelles de recherche ont suscité un grand intérêt au sein des communautés scientifiques d'autres pays qui envisagent une participation scientifique éventuelle avec l'ILL. La participation scientifique de l'Espagne à l'ILL est devenue effective au début de l'année 1987 et les négociations en vue d'une participation de la Suisse sont presque terminées.

L'installation centrale de l'ILL est le réacteur à haut flux, qui, depuis le programme de modernisation fonctionne de manière plus fiable que jamais. Fin 1987 la nouvelle source froide horizontale a pu être installée et testée comme prévu. Nous adressons notre reconnaissance toute particulière aux techniciens et ingénieurs concernés pour cette réalisation exceptionnelle.

La conception et la construction des instruments pour la deuxième source froide sont bien avancées. L'installation du spectromètre 3 axes IN14 est presque achevée, ainsi que celle de l'expérience (n,n̄), qui résulte d'une collaboration des Universités de Padoue, Pavie, Heidelberg et de l'ILL. Les projets IN15, spectromètre à écho de spin à haute résolution et IN10C, spectromètre à rétrodiffusion à haute intensité sont à un stade de projet avancé. Il a également été décidé de construire un nouvel instrument de diffusion aux petits angles D22. Bien qu'ainsi 4 nouveaux instruments soient en projet ou en construction, des efforts supplémentaires dans le domaine du développement des méthodes sont rendus nécessaires par l'apparition de nouvelles applications de la diffusion neutronique, comme par exemple l'application de la réflexion totale dans l'étude des couches minces ou l'utilisation de la spectroscopie de Brillouin pour étudier la dynamique de systèmes désordonnés. Dans cette optique, l'apport des expériences spéciales dites "S" représente une aide précieuse.

Finalement la décision de renouveler l'ordinateur central aura pour conséquence une amélioration substantielle de l'acquisition et du traitement des données, ce qui est d'une grande importance pour toutes les expériences.

Malgré les limitations dues aux travaux liés au projet de la deuxième source froide, 5 cycles de réacteur ont pu avoir lieu en 1987, conformément au planning. Sur un nombre total de 1056 propositions d'expériences soumises, ce qui correspond à une demande de temps de faisceau de 10497 jours, 742 expériences ont pu être réalisées, soit un temps de faisceau total de 5347 jours.

## Programme scientifique et résultats

Les travaux scientifiques réalisés en 1987 et les résultats obtenus sont repris plus en détail dans les rapports des Collèges. Je me contenterai ici de résumer quelques résultats particulièrement intéressants.

Après la découverte des nouveaux supraconducteurs à haute température par Bednorz et Müller, il était clair que la diffu-

sion des neutrons pourrait aider à clarifier leurs propriétés microscopiques. Exceptionnellement et à court terme, un temps de faisceau d'environ 100 jours sur différents instruments a été attribué pour l'étude de ces problèmes. L'ILL a ainsi pu apporter une contribution significative ; c'est ainsi que, pour la première fois, il a été possible de déterminer la structure du supraconducteur "100 K",  $\text{YBa}_2\text{Cu}_3\text{O}_7$ , et en particulier la configuration des défauts d'oxygène. Une diffusion de neutrons polarisés sur cet alliage et une analyse de polarisation ont montré que la diffusion magnétique totale est extrêmement faible, et que, par conséquent, des mécanismes de couplage magnétique étaient improbables. Par contre on a découvert, dans  $\text{GdBa}_2\text{Cu}_3\text{O}_7$  par exemple, un ordre magnétique à basse température. Enfin, on dispose maintenant des résultats de toute une série d'études dynamiques de réseau de  $\text{YBa}_2\text{Cu}_3\text{O}_{7-x}$ .

Outre ce programme ad hoc, un nombre considérable de résultats excellents ont été obtenus au cours des programmes de mesure normaux, par exemple

- sur les excitations magnétiques à haute énergie dans les métaux de transition 3d (source chaude) et sur les excitations de faible énergie dans les grenats antiferromagnétiques (source froide)
- dans les études de la structure quasi-cristalline des alliages Al-Li-Cu et sur la structure des hydrates d'hélium
- dans les systèmes désordonnés sur la dynamique de la transition vitreuse et sur le spectre d'excitation de mélanges de  $^3\text{He}$  et  $^4\text{He}$
- dans les systèmes biologiques grâce à la méthode de variations de contraste sur le comportement du polymérase RNA lors de la transcription
- dans le domaine de la chimie, il faut citer les mesures de réflexion de surfaces sur des solutions de polymères et la preuve faite pour la première fois de l'existence de modes de relaxation de forme de gouttelettes dans des microémulsions
- dans les travaux relatifs à la physique nucléaire et à la physique des particules, il convient de mentionner en particulier la découverte de nouveaux produits de fission riches en neutrons, tels que  $^{76}\text{Ni}$  (LOHENGRIN) et les premières déterminations de durées de vie extrêmement courtes avec le spectromètre à double cristal GAMS4.

## Questions de Personnel

L'année écoulée n'a, dans l'ensemble, pas apporté de grands changements de personnel. Il faut mentionner que A. Heidemann a succédé à C. Janot à la tête du département EDEX.

En 1987 également, des membres de l'Institut ont reçu des prix ou des distinctions. D. Richter a été honoré du prix Walter Schottky pour la physique de l'état solide par la Société Allemande de Physique. W. Stirling a accepté un poste de Professeur titulaire à l'Université de Keele. O. Schärpf a accepté une chaire à l'Université Technique de Munich à titre provisoire. Ces distinctions sont considérées comme un grand honneur pour l'Institut tout entier.

Pendant la période du rapport, un certain nombre de personnalités bien connues du monde politique et scientifique ont à nouveau visité l'ILL. Outre les participants aux célébrations du 20<sup>e</sup> anniversaire, il faut citer entre autres la visite d'une délégation du gouvernement chinois conduite par le Ministre chinois de la Recherche, M. Song Gian, ainsi que la visite du vice-président de la Commission de la Communauté Européenne, K.-H. Narjes.

## Collaboration avec l'ESRF

Avec la décision d'établir la Source Européenne de Rayonnement Synchrotron dans le voisinage immédiat de l'ILL, les conditions idéales ont été réunies pour la création d'un centre unique pour la recherche sur la matière condensée.

Pendant la phase préparatoire de l'ESRF, commencée en 1986 et poursuivie en 1987, l'ILL a reçu le mandat de représenter l'ESRF jusqu'à sa création légale, et a assisté l'équipe chargée de la mise en place dans de nombreux domaines grâce à ses installations et services. Des progrès considérables ont été faits en 1987 en ce qui concerne la préparation de la future collaboration entre les deux instituts. En particulier, un accord a été obtenu quant à la création d'un site de recherche intégré commun, ce qui est une condition essentielle pour une coopération économique et effective, et qui devrait se baser sur la signature avec la CEA d'un nouveau bail commun.

Il faut noter que l'ILL a maintenant libéré une partie du site qui se trouvait dans la future zone ESRF et que maintenant le financement des travaux d'évacuation du reste du site est assuré.

Un certain nombre de groupes de travail communs ont été créés dans le but d'examiner en détail les possibilités concrètes d'une future coopération dans les domaines scientifiques, techniques et administratifs, et leur travail a déjà conduit à quelques propositions intéressantes qui devront être prises en compte lors des projets de construction et de financement. Il reste à souhaiter que l'ESRF obtienne le plus tôt possible un statut légal avec la signature d'un accord intergouvernemental.

*J'ai le triste devoir de vous faire part du décès de notre ancien collègue Stuart Wilson. Pendant de nombreuses années M. Wilson, en tant que responsable du diffractomètre D16, a été un membre actif du groupe cristallographie. Il avait pris au printemps de cette année une retraite bien méritée. Nous garderons de lui un excellent souvenir.*

W. Gläser

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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 est la suivante :

Collège 2 : théorie.

Collège 3 : physique fondamentale et nucléaire.

Collège 4 : excitations structurales 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 of the College

E. Akkermans  
H. Capellmann  
P. Grangé  
D. Grempel  
J. Jolie  
A. Maggs  
P. Nozières  
S. Robaszkiewicz  
J. Rodriguez  
N. Schopohl  
T. Sluckin  
J. Solyom  
P. Thalmeier

## Long Term Visitors

J. José  
V. Marchenko  
R. Prange  
L. Sneddon  
J. Vannimenus  
M. Walker  
P. Wiegmann  
D. Wolff

## Introduction

Research within the Theory College covered many topics ranging from condensed matter physics (crystal growth, fluids, liquid crystals, heavy fermions, high  $T_c$  superconductivity, disorder and localization, nonlinear systems, magnetic chains) to nuclear physics (symmetries, energy spectra, Landau Vlasov transport theory, microscopic calculations of the nuclear mean field). Some of those activities were carried out in close collaboration with experimental groups at the ILL. The scientific life within the College also profited from many short, medium and long term visitors.

## Scientific Trends and Highlights in 1987

### High Temperature Superconductivity

The advent of "High  $T_c$ " superconductivity in Cu-oxides had its impact also within the theory college. S. Robaszkiewicz had already been working on a theory of superconductivity based on local electron pairing (e.g. bipolaronic superconductivity) before the LaBaCu oxides and the YBaCu oxides became "hot" (together with Alexandrow, Micnas, Ranninger). The theory had been developed for BaBiPb oxides, a superconductor now recognized to have many similarities to the Cu oxides. An application of the theory to the new materials was carried out. Unfortunately S. Robaszkiewicz had to leave the theory college at the end of February 87 to go back to Poland, right at the time when the subject really "took off".

H. Capellmann collaborated with T. Brückel and O. Schärpf in experimental neutron scattering studies of  $YBa_2Cu_3O_7$  using polarization analysis. The results indicate that the total magnetic scattering intensity is vanishingly small, making magnetic couplings (the basis for many "modern" theories) as a source for superconductivity in  $YBa_2Cu_3O_7$  highly unlikely.

N. Schopohl (together with A. Baratoff, IBM Zürich) studied the penetration depth for anisotropic superconductors within the Landau theory. Having "high  $T_c$ " in mind, J. Rodriguez started a study of magnetic defects in two-dimensional antiferromagnets.

### Heavy Fermions and Intermediate Valency

P. Thalmeier studied elastic and magneto-acoustic phenomena in heavy fermion systems (elastic constants of  $CeCu_6$ ,  $CeRu_2Si_2$ ) and dilute "Kondo" systems (magneto-acoustic quantum oscillations in  $(CeLa)B_6$ ). Furthermore he investigated the influence of quasiparticle scattering on the de Haas van Alphen amplitudes. To understand the superconductivity of heavy fermion systems, J. Rodriguez examined superconducting groundstates with vector or quadrupolar order parameters and their collective excitations. He also studied the effects of impurities on superconductors with exotic order. H. Capellmann continued theoretical studies of couplings between magnetic, charge and lattice fluctuations. A theory explaining observed electrical resistivity anomalies in terms of charge fluctuation effects was developed. He extended the close experimental collaboration with K. Neumann, O. Schärpf and K. Ziebeck to explore magnetic fluctuations and lattice properties of heavy fermion systems.

### Fluids, Liquid Crystals, Colloidal Systems

P. Nozières calculated the Onsager coefficients coupling convection and sedimentation in suspensions to first order in concentration. He thus verified predictions based on Onsager symmetry arguments and also obtained coupling terms due to concentration gradients.

N. Schopohl and T. Sluckin studied the structure of defects in nematic liquid crystals. The types of defects treated were disclination lines and point defects. T. Sluckin examined the phase diagram of charged colloidal systems and models of polydisperse fluids with variable polydisperse distributions. He looked at aspects of the equilibrium statistical mechanics of liquid crystals with particular emphasis on wetting and layering in smectic phases. T. Sluckin and T. Ziman completed a study of two-dimensional liquid crystals which also exhibit magnetic behaviour.

A. Maggs' interest was the very rich phase behaviour of systems containing amphiphilic molecules, in particular ternary oil-water-surfactant solutions and solutions of biological lipids. He developed a theory to explain observations of phases of cubic symmetry, which occur at high concentration of amphiphile causing the molecules to organize into surfaces which then organize further into the observed mesostructures.

### Transport in Disordered Systems, Localization

D. Grempel studied magnetic couplings between conduction electrons and impurities and the arising consequences for transport coefficients in disordered media. In particular effects on the metal insulator transition, the spin relaxation rate and the crossover between regimes of weak and strong magnetic coupling were addressed. In collaboration with S. Fishman and R. Prange, D. Grempel had shown that quantum interference effects responsible for the Anderson transition in random media have an analogy in the quantum

mechanics of non-linear systems: near a critical point where the motion of a classical non-linear system becomes chaotic, quantum corrections lead to interference, preventing the transition to occur. A renormalization group study of these quantum correction was carried out.

E. Akkermans' activities essentially focussed on transport properties in disordered media. He studied a model to explain quantitatively experimental results on the coherent backscattering effect (weak localization). Furthermore, he examined properties near the localization transition, the universal conductance fluctuations in disordered media and further cumulants in order to obtain the distribution function of the conductance. L. Sneddon studied "mode locking", a strongly nonlinear effect observed in the conductivity of charge density wave systems. He performed simulations finding that mode locking does occur in classical systems, because of resonance effects between the ac part of the applied field and the fluctuating pinning potential seen by the charge density wave. These results corrected assumptions that mode locking is intrinsically a quantum effect. Sneddon also continued work on the critical properties of incommensurate systems.

P. Grange's activities dealt with calculations of the nuclear mean field and corrections thereto and with Landau Vlassov transport theory. Zero and finite temperature contributions of groundstate correlations to the nuclear mean field were studied in nuclear matter at normal densities. The initial framework was non-relativistic; extensions to include relativistic corrections were examined and the necessity of a quantum relativistic nuclear transport theory discussed.

Secretary: H. Capellmann

## Various Activities in Condensed Matter Physics

J. Solyom's attention focussed on properties of one-dimensional magnetic systems. He showed that for spin values higher than  $1/2$  spontaneous dimerization can only occur for small elastic moduli, in contrast to spin  $1/2$  chains where dimerization occurs independently of the strength of the elastic moduli. General isotropic magnetic interactions including bilinear and biquadratic terms were studied. The phase diagram and excitation spectrum of the  $S = 1$  model was determined. Together with E. Niebur, Solyom studied various improvements of the mean field like renormalization group approach.

P. Nozières and D. Wolf studied problems of interfaces and crystalline growth in particular the interplay between elasticity and capillarity. Another problem treated was the growth of a surface in the diffusive region (when diffusion of atoms from one point of the surface to another is important).

N. Schopohl in collaboration with D. Waxman studied the A-B phase boundary of superfluid  $^3\text{He}$ : the theory shows that bound quasiparticle states exist in the interface region. Their influence on thermal resistance, transmission and reflection coefficients, and local density of states was examined.

D. Gempel treated non-linear instabilities in chemical systems, in particular competitive growth of particles in a sol and the resulting spontaneous precipitation patterns. The length scale selection hereby is a purely non-linear effect, the dependence of these lengthscales on the parameters of the solution in its initial state was studied.

## Nuclear Physics

J. Jolie's work during 87 can be divided into three major parts. Firstly he continued earlier work on dynamical symmetries and supersymmetries. Secondly, he calculated the energy spectra of nuclei which were measured at the ILL in different models, this in collaboration with the College 3 members. Thirdly, he developed a method to analyze data taken on GAMS-4 which allows the determination of lifetimes of nuclear excited levels, this again in close collaboration with College 3 members.

# COLLEGE 3 NUCLEAR AND FUNDAMENTAL PHYSICS

## Members of the College

### I. Internal Members

J.P. Bocquet  
H.G. Börner  
J. Copnell  
W. Drexel  
D. Dubbers  
H.R. Faust  
I. Förster  
P. Geltenbort  
F. Hoyler (1.4.87)  
B. Krusche  
W. Lippert  
W. Mampe  
R.A. Oliver  
D. Richardson  
S. Robinson  
K. Schreckenbach  
U. Stöhlker (1.9.87)  
H. Weikard (1.2.87)

### II. Long Term Visitors

W. Andrejtscheff (Belgrade)  
G. Azuelos (Triumph)  
F. Baumann (Bayreuth)  
T. Bitter (Heidelberg)  
L. Bondarenko (Kurchatov)

R. Brissot (CENG)  
G. Danilyan (ITEP)  
A. D'Eer (SCK/CEN)  
M.S. Dewey (NBS)  
R. Gähler (TUM)  
/R. Golub (HMI)  
G. Greene (NBS)  
P. Grivot (ISN)  
E. Grüber (Vienna)  
E. Gutmiedl (TUM)  
E. Kessler (NBS)  
I.A. Kilvington (RAL)  
R. Kossakowski  
(Chambery)  
E. Krüger (PTB)  
S. Lamoreaux (Seattle)  
P. Liaud (Chambery)  
W. Nistler (PTB)  
M. Pendlebury (Sussex)  
V. Rabbel (Giessen)  
A. Steyerl (TUM)  
D. Thompson (RAL)  
P. Vittoz (Grenoble)  
G. Vivier (Grenoble)  
W. Weirauch (PTB)  
A. Zeilinger (Vienna)

## Collaborations of College 3 with Institutes and Universities in 1987

Bayreuth  
Belgrade  
Berlin HMI  
Bonn  
Bordeaux CEN  
Braunschweig PTB  
Braunschweig Univ.  
Brookhaven Nat. Lab.  
Bruyere-Le-Chatel  
Chambery  
Darmstadt GSI  
Fribourg  
Geel JRC  
Giessen  
Göttingen  
Grenoble CEN  
Grenoble ISN  
Grenoble Univ.  
Harvard  
Harwell  
Heidelberg

Jülich KFA  
Karlsruhe KFK  
Köln  
Leningrad NPI  
Livermore LL  
Mainz  
Manchester  
Mol SCK/CEN  
Moscow ITEP  
Moscow Kurchatov  
München TU  
München Univ.  
Rutherford Lab.  
Seattle  
Sofia  
Surrey  
Sussex  
Tübingen  
Utah St. Univ.  
Vienna Atominst.  
Washington NBS

## General Summary

The activities of College 3 cover a broad spectrum of interest involving various aspects of the fundamental interactions which govern the structure of the atom, its nucleus and its constituent particles.

Nuclear structure studies continued, using the electron spectrometer BILL (PN2), the gamma ray crystal spectrometers GAMS-1, -2/3 (PN3) and GAMS-4, the pair spectrometer PN4 and the on-line separator OSTIS (SN6). That the ILL is still at the forefront of neutron capture spectroscopy was demonstrated by the large number of contributions to the 6th International Symposium on Capture Gamma-Ray Spectroscopy (held in Leuven, Belgium in 1987) which related to results obtained using ILL facilities.

The thermal neutron induced fission process is studied using the parabolic spectrometer LOHENGRIN (PN1) and the energy-time-of-flight spectrometer COSI FAN TUTTE (PN8). Of particular note in this field are the studies of extremely rare fission processes and the identification of exotic new isotopes made possible by the development of a big ionisation chamber (BIC) which makes possible the measuring of masses at detection rates as low as one fission product per day.

The field of fundamental physics at the ILL concerns itself mainly with investigations of the basic properties of the free neutron such as the search for a possible electric charge or electric dipole moment and the accurate determination of the parameters which describe neutron  $\beta$ -decay. These studies make great use of the facilities provided by SN5, the ultra cold neutron - very cold neutron (UCN - VCN) installation on level D and the polarized beam SN7.

In addition to the instruments mentioned above several other neutron beams are used by College 3 to study such topics as neutron induced fission and charged particle evaporation cross sections, activation analysis of geological and biological materials and gamma-gamma coincidence and correlation studies of nuclei following thermal neutron capture.

## Scientific Trends and Highlights in 1987

### Nuclear Structure

With the departure of G. Colvin (1.11.86) and F. Hoyler (1.4.87) the nuclear spectroscopy group suffered a temporary 40 % depletion in manpower - a situation which continued throughout the year. Despite this a full and wide-ranging program was maintained concentrating, as usual, mainly on nuclear structure investigations but also encompassing aspects of both solid state and fundamental physics.

With the increasing ability of nuclear models to successfully describe general nuclear characteristics the experimental focus has begun to shift to the study of particular transitions selected for their ability to discriminate between different model predictions.

A major topic in nuclear spectroscopy at the conversion electron spectrometer BILL (PN2) was the study of F-spin (isospin for a system of proton and neutron bosons) forbidden M1 transitions between collective levels. (Collaboration with Institut für Kernphysik, Köln). Measurements were made of E2/M1

admixture for a number of  $I \rightarrow I-1$  transitions in the gamma bands of  $^{178}\text{Hf}$ ,  $^{162}\text{Dy}$  and  $^{188}\text{Os}$ . These transitions are very weak and usually occur in regions of the spectrum with high line density so that very good resolution and statistics are needed. Fig. 1 shows an example from the reaction  $^{161}\text{Dy}(n,e^-)$  which demonstrates the high dynamic range of BILL. The transitions of interest are the  $L_{II}$  and  $L_{III}$  subshell conversion lines of the 98.054 keV transition ( $4^+ \rightarrow 3^+$ ).

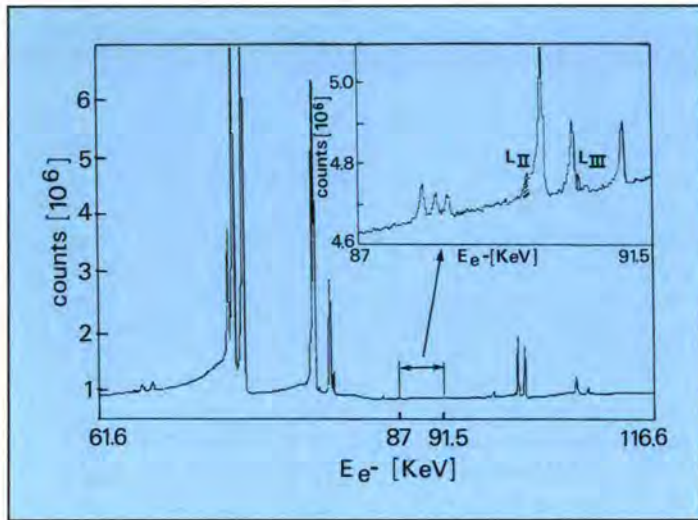


Figure 1: Part of the electron spectrum from the  $^{161}\text{Dy}(n,e^-)$  reaction obtained with the BILL spectrometer showing the spectrum between 61.6 and 116.6 keV, measured 30s/point. The projected inset with the  $L_{II}$  and  $L_{III}$  conversion lines of the 98.054 keV  $\gamma$ -transition was obtained by scanning many times (total measuring time 255s/point).

A long standing effort at PN2 is the study of E0 transitions. For transitions between  $0^+$  states whose energies differ by less than  $2m_e c^2$  internal conversion is the only decay channel and the relative intensities of the conversion lines provide information on the nuclear wave functions. One example of this comes from the study of  $^{196}\text{Pt}$  which is thought to be a good example of the O(6) limit of the interacting boson model (IBM) based on earlier  $(n,\gamma)$  studies and measurements of E0 transitions to the ground state. However, now a strong  $0_3^+ - 0_2^+$  E0 transition has been observed which should be forbidden in the O(6) picture. (Collaboration with Sussex Univ.).

For energies larger than  $2m_e c^2$  a second branch becomes available for E0 decay - namely internal pair production. Up to now experimental measurements of the ratio of the two competing processes were only reported for light nuclei with a precision of typically 20 %. Therefore this ratio was measured for the strong 1306 keV E0 transition in  $^{114}\text{Cd}$  by comparing the intensity of the conversion line with that of the corresponding  $e^+$  spectrum from internal pair production. A final precision of about 5 % is expected.

In the continued close partnership between ILL experiments and tests of the IBM detailed spectroscopy has been undertaken on  $^{154}\text{Sm}$  (following double neutron capture in  $^{152}\text{Sm}$ ) and  $^{188}\text{Os}$  (Collaborations with Univ. of Tübingen and Univ. of Manchester). Interest in the former centres around the properties of the third  $K = 0^+$  band and an attempt to establish its systematic behaviour in many rare earth nuclei, thus helping to distinguish between the various theoretical explanations proposed. The  $^{187}\text{Os}(n,\gamma)$  experiment performed at the bent crystal spectrometers GAMS 1,2/3 (PN3) was distinguished by the excellent angular resolution achieved of 0.35 arc-seconds which represents the best ever obtained with bent quartz crystals under realistic measuring conditions.

As already mentioned in last years report the study of odd-odd nuclei is becoming increasingly important since they provide a laboratory for the study of the residual proton-neutron interaction. The completion of a study of  $^{76}\text{As}$  has produced a level scheme which can be approximately reproduced within the framework of a supersymmetric multiplet. Due to their high line density few doubly odd nuclei in the actinide region have been investigated in detail. Efforts in this region have continued with the evaluation of data obtained following neutron capture in  $^{241}\text{Am}$  (Fribourg, Livermore, ILL collaboration). A detailed level scheme for  $^{242}\text{Am}$  has been constructed (Fig. 2) including six new well established bands and several Gallagher-Moszkowski doublets. Attempts to interpret this nucleus have been made using the strong coupling model and a technique based on the level structure of adjacent odd-A nuclei.

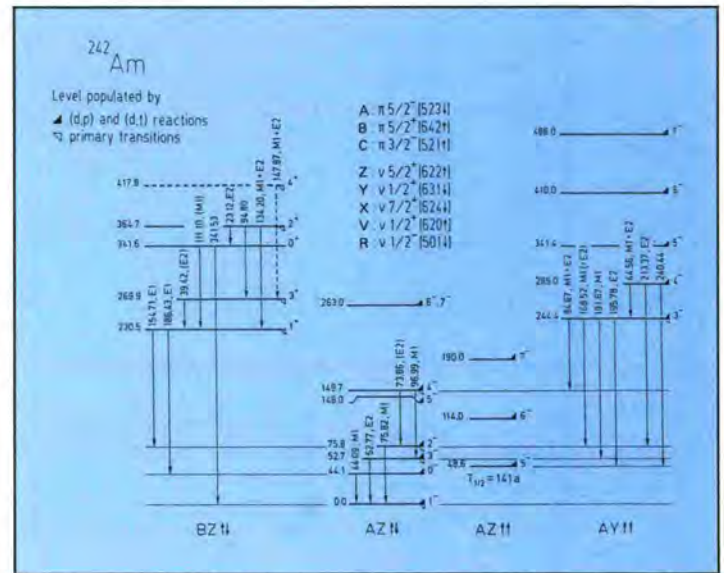


Figure 2: Portion of the level scheme obtained for the odd-odd nucleus  $^{242}\text{Am}$ . In total 12 rotational bands have been observed built on the proton and neutron configurations shown in the upper part of the figure and including five Gallagher-Moszkowski pairs.

The study of more general nuclear properties such as level densities and integrated transition strengths provide a tool for the study of statistical aspects of nuclear structure. The pair spectrometer, PN4, has been used to provide such data for the nuclei  $^{88}\text{Sr}$  and  $^{32}\text{P}$ . (Collaboration with Univ. of Göttingen). In addition, studies of the odd-A nuclei  $^{153,157}\text{Gd}$  incorporating data taken with PN4 have resulted in level schemes which show reasonable agreement with Nilsson model predictions. (Collaboration with SCK/CEN Mol).

It is generally agreed that a sensitive test of nuclear models is the correct prediction of absolute transition rates from excited levels. One common method to determine these values experimentally is to measure the lifetime of these states. The Doppler Shift Attenuation Method (DSAM) has been successfully applied to charged particle induced heavy ion reactions since the advent of high resolution germanium detectors. In such reactions the recoil velocities are typically of the order of 0.1 % - 1 % of the velocity of light. Recently it has been demonstrated at ILL that high resolution  $(n,\gamma)$  spectroscopy can be used for the determination of such lifetimes through the observation of Doppler Broadening. The broadening of these (secondary)  $\gamma$ -rays is due to the recoil imparted by the feeding transitions. The recoil velocity thus obtained is  $10^{-4}$  to  $10^{-5} c$  (cf. DSAM). Only the high resolving power

( $\Delta E/E \sim 10^{-5}$ ) obtained with the two axis flat crystal spectrometer GAMS-4 allows the direct observation of these effects. Using this technique Doppler broadened secondary transitions have been observed following thermal neutron capture in  $^{53}\text{Cr}$ . (Collaboration with Univ. of Göttingen). The lifetime of the 3074 keV  $2_3^+$  state in  $^{54}\text{Cr}$  has been determined, from the depopulating 2239 keV transition (Fig. 3), to be  $\tau = 17(8)\text{fs}$ . The resulting large M1 strength to the  $2_1^+$  state ( $B(M1) = 0.29(+27,-9)\mu_N^2$ ) suggests that the  $2_3^+$  state is of predominantly mixed symmetry character within an IBM-2 description. Additional lifetimes have been determined in  $^{49}\text{Ti}$  and  $^{57}\text{Fe}$ .

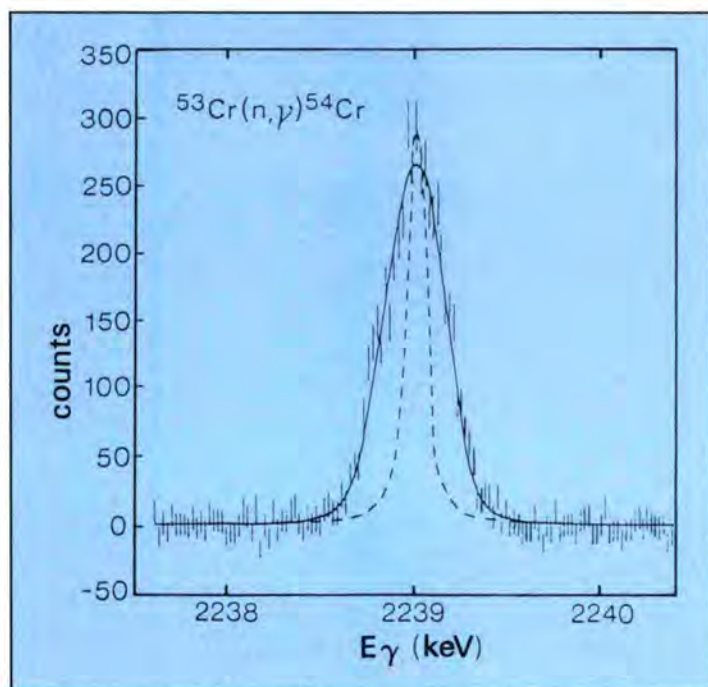


Figure 3: Doppler broadened line shape of the 3074  $\rightarrow$  835 keV transition in  $^{54}\text{Cr}$  observed in first reflection order after the reaction  $^{53}\text{Cr}(n,\gamma)^{54}\text{Cr}$ . Also given are the spectrometer function (dashed line) and the fitted line shapes (solid line).

The above procedure can also be turned around and a known lifetime used to study the stopping process of ions/atoms travelling in different solid state environments. This has been done by studying the Doppler broadened shape of the well known 478 keV ray emitted from the recoiling  $^7\text{Li}$  nucleus after the  $^{10}\text{B}(n,\alpha)$  reaction, using the GAMS-2/3 spectrometers. (Collaboration with Univ. of Köln and Utah State Univ.).

The origin of narrow positron lines observed in heavy ion collisions at the UNILAC of the GSI (Darmstadt) still remains an unsolved puzzle. The observation of  $180^\circ$  (center of mass) correlated  $e^+e^-$  emission points towards the creation of a hypothetical  $(e^+e^-)$  state which decays into an  $e^+e^-$  pair with a lifetime longer than the collision time. In the reverse reaction such a state may manifest itself as a resonance in the excitation function of  $e^+e^-$  Bhabba scattering. The conversion electron spectrometer BILL offers an excellent opportunity to study this excitation function by acting as a variable energy positron source. Compared to a first test run in 1986 experimental conditions have been considerably improved (See also technical section of this report). In a first measurement in spring 1987 (Collaboration with GSI Darmstadt) a 45 mm  $\times$  90 mm  $\times$  2 mm Ti plate was used as an in pile target. The emitted positrons were momentum analysed by the spec-

trometer and focussed on the scattering foil. A beam intensity of  $8 \times 10^4 e^+/\text{sec}$  on an area of 10 mm  $\times$  25 mm was achieved. Scattering from a mylar foil ( $\text{C}_{10}\text{H}_8\text{O}_4$ ) and a metallic thorium foil was studied in the energy range from 1 to 2 MeV by a four detector arrangement. The measured ratio of Bhabba to Mott scattering as a function of the bombarding energy is shown in Fig. 4. Within the statistical uncertainty of approximately 2.5 % for the mylar and 3-4 % for the thorium all data points are in agreement with a smooth curve at the  $3\sigma$  limit. Since this run the usable beam intensity has been improved by employing a thicker Ti plate and an 8 detector arrangement. This allowed the study of scattering in a Beryllium foil segment of 20 mm  $\times$  100 mm without a loss in energy resolution and gave an increase in recorded event yield by about an order of magnitude with 15000 Bhabba events per hour. This data is currently under evaluation and a statistical accuracy of better than 1 % is expected.

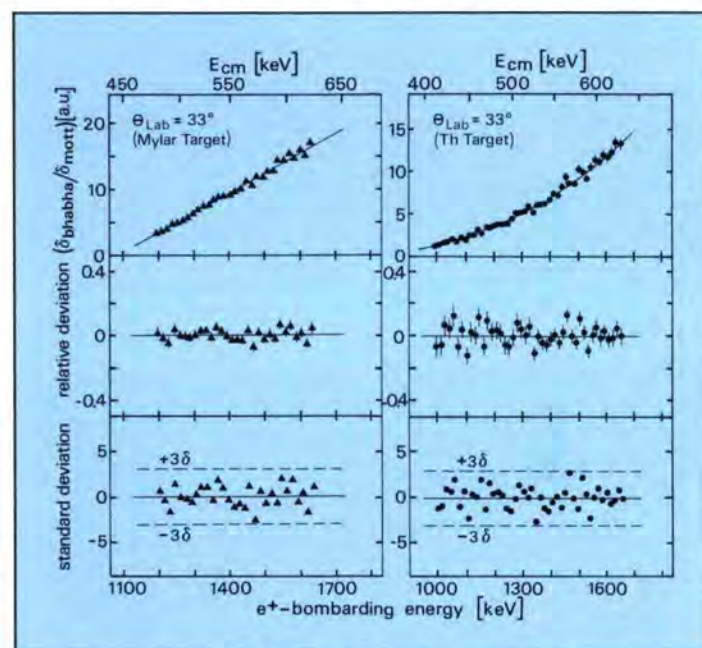


Figure 4: Measured ratio of Bhabba to Mott scattering as a function of the bombarding energy (uppermost part) for mylar (left) as well as for thorium (right) targets. Also shown are the relative (middle) as well as the standard (lowest part) deviations from the expected smooth distribution fitted to the data points (solid line in the uppermost part).

The strength of the mass separator OSTIS (SN6) since its installation at the ILL in 1975 has been the steady accumulation of data concerning the systematics of nuclear structure information as a function of both N and Z. The instrument has produced important results concerning nuclear structure such as the location of shape transitions, shell gaps, shape coexistence,  $\beta$ -decay systematics and information concerning  $\beta$ -delayed neutron emission. Much of this information is not only relevant for the testing of various nuclear models but also as input for astrophysical nucleosynthesis calculations and the study of reactor technology (particularly in the study of fast breeder reactors). The results from experiments performed at OSTIS have made significant contributions to all these fields. However the consensus of the collaborators who support this instrument is that the program of experiments which can be performed is very near to completion, and so it has been mutually agreed that OSTIS will no longer be available for use as from the end of 1987.

Results from OSTIS this year have included the complete range of studies mentioned above such as  $\beta$ -delayed neutron studies on Br and I isotopes and the measurement of first excited state lifetimes in  $^{97,98,99}\text{Sr}$  and  $^{99}\text{Y}$  (Collaboration with Mainz and KFA Jülich). First results indicate a stronger deformation in  $^{98}\text{Sr}$  than in  $^{100}\text{Sr}$ . Also,  $Q_\beta$  values have been determined for the medium to low yield nuclei  $^{87-89}\text{Br}$  (Collaboration with T.U. Braunschweig)  $^{128,130,132}\text{In}$  and  $^{136-140}\text{I}$  (Collaboration with Physikal. Inst. Giessen). Finally the systematic study of the  $N = 84$  isotones has been extended to  $^{138}\text{Xe}$  with the first attempt to measure spins and M1/E2 mixing ratios in this nucleus. The preliminary results show good agreement with IBM-2 calculations and suggest that the  $2_2^+$  state is of almost pure mixed symmetry character.

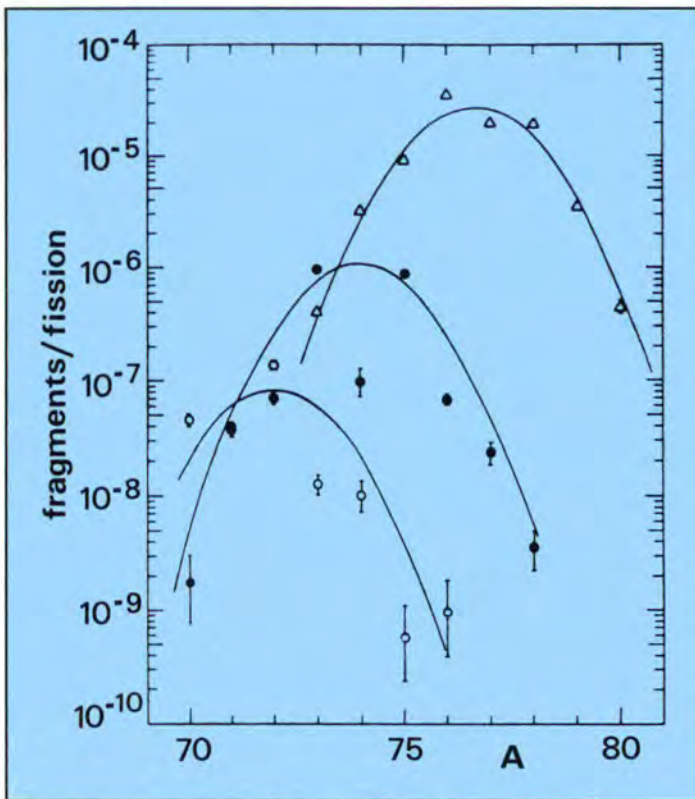


Figure 5: Fission rates (fragment/fission) for selected  $A/q$  and  $E$  values ( $A = 70-80$ ). The continuous line is a Gaussian curve drawn to guide the eye. The width parameter is 3 a.m.u.(FWHM), a typical value for isotopic distributions in fission.  $\Delta$ Zn,  $\bullet$  Cu,  $\circ$  Ni. The count rate of  $10^9$  fragments/fission corresponds to a count rate of 1 fragment/day in the ionization chamber.

## Fission

Though the neutron induced fission process is literally at the heart of the ILL the fission mechanism itself is far from being well understood. The main task of the fission group is to gather systematic information on how the distribution of fission fragments depends on the nuclear parameters of the fissioning system and of the fission products themselves.

Most of the experiments performed on Lohengrin (PN1) this year have made use of the improved Big Ionisation Chamber (BIC - see technical section) to study exotic processes in nuclear fission. Some of these experiments have as their aim the study of the low fragment yield far asymmetric region between Co and Se. Measurements of the kinetic energy dependence of nuclear mass and charge distributions for the  $^{235}\text{U}(n_{th},f)$  system are almost complete. Fig. 5 shows the nuclear charge

distribution for Zn, Cu and Ni below  $A = 86$ . Besides the obvious sharp drop in yields with decreasing mass a pronounced odd-even staggering is apparent for Cu and Ni. Similar studies have also been started for the  $^{241}\text{Am}(2n_{th},f)$  and  $^{239}\text{Pu}(n_{th},f)$  systems.

Experiments in the symmetric mass region for the  $^{241}\text{Am}(2n_{th},f)$  system have continued. The ionic charge state and kinetic energy dependence of the mass distribution has shed new light on the fission mechanism for near symmetric mass splits. While, just above the symmetric mass, there is a considerable fission product yield the expected complementary yield below symmetry is missing. This leads to the idea that symmetric fission does not produce a symmetric share of excitation energy; just below symmetry the fragments have a large degree of internal excitation which they lose by neutron evaporation - thus depleting the yield in this mass region (Fig. 6).

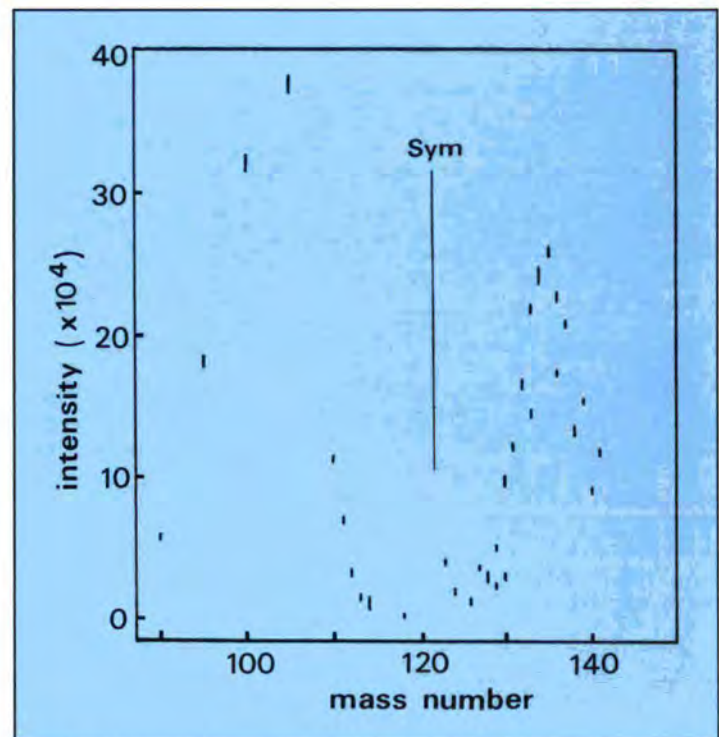


Figure 6: Intensity distribution of fission products with near symmetric mass splits for the reaction  $^{241}\text{Am}(2n,f)$  for selected charge states

The measurement of the initial angular momentum of the fragments via the subsequent population of excited high spin states showed another anomaly for masses near symmetry; namely a considerable drop in angular momentum for  $^{132}\text{Sn}$ . A systematic investigation of angular momentum created in the fission of  $^{233}\text{U}$ ,  $^{235}\text{U}$  and  $^{239}\text{Pu}$  has shown that all three systems behave similarly in the population of isomeric states of different spin in the fission products (Fig. 7).

With the improvement of the BIC the search for new exotic isotopes has begun, initially in the thermal neutron induced fission of  $^{235}\text{U}$ . In the neutron rich region around the  $Z = 28$  closed shell, the r-process nuclei  $^{70-74}\text{Ni}$  have been observed for the first time, together with the new nuclei  $^{74-77}\text{Cu}$ . (See blue box contribution). Preliminary data analysis following a search for heavy cluster fission decay modes has shown indications that  $^{28}\text{Mg}$  is produced in the fission of  $^{233}\text{U}$ .

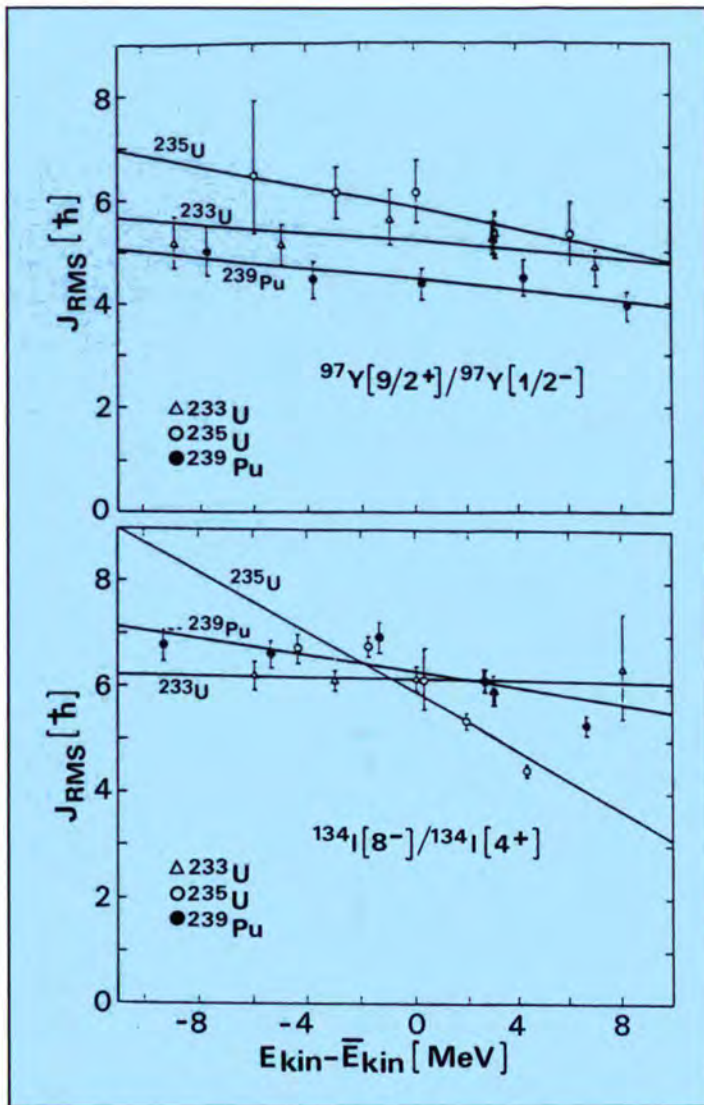


Figure 7: Mean angular momentum created in the fission process as a function of the kinetic energy for selected fragments. For the three systems  $^{233}\text{U}$ ,  $^{235}\text{U}$  and  $^{239}\text{Pu}$  the mean, angular momentum amounts to 5-7  $\hbar$ , almost independent of the kinetic energy value

However the kinetic energy distribution for this nucleus suggests that it comes from a tripartition channel.

A long standing search for masses  $A < 21$  produced in tripartition decay has continued with the first observation of  $^{21}\text{Ne}$  emission. Besides a pronounced proton odd-even effect in the yields it was found that for light particles with  $Z > 6$  the mean kinetic energy does not change appreciably with increasing proton number.

The analysis of the mass-, energy- and charge characteristics of the  $^{241}\text{Pu}(n_{\text{th}},f)$  fragments measured at the coincidence fission fragment spectrometer COSI FAN TUTTE (PN8) has been completed. The total nuclear charge distribution for the light fragments is shown in Fig. 8 from which a proton odd-even effect of 9(1)% has been calculated. This shows similar behaviour to that of the  $^{239}\text{Pu}(n_{\text{th}},f)$  system obtained in an earlier experiment at PN1, for which a value of 11(1)% was reported. This value fits well into the systematics of proton odd-even effects as a function of  $Z_F^2/A_F$  (where  $Z_F$  and  $A_F$  are the charge and mass of the compound nucleus). This proton odd-even effect is clearly demonstrated in Fig. 9 which

shows the correlation between the standard deviations  $\sigma_Z$  and the average nuclear charge  $Z$  of the isobaric nuclear charge distribution.

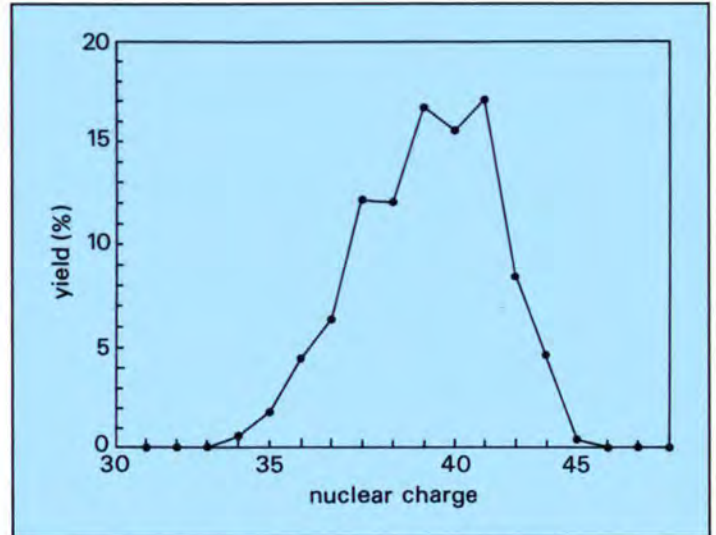


Figure 8: Total nuclear charge distribution for the light fragment group in the thermal neutron induced fission of  $^{241}\text{Pu}$ .

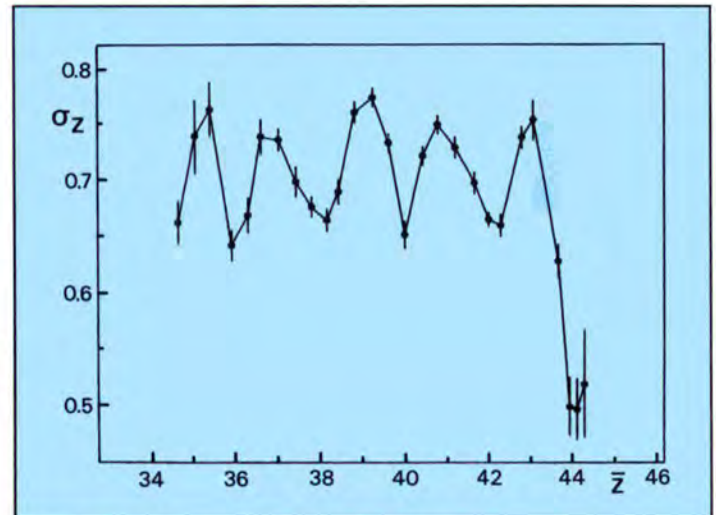


Figure 9: Standard deviations  $\sigma_Z$  of the isobaric charge distribution as a function of the average isobaric nuclear charge  $\bar{Z}$

Measurements on the  $^{249}\text{Cf}(n_{\text{th}},f)$  system have also recently been completed. In total more than two million fission events were recorded and a detailed analysis is underway. Mass yields and kinetic energies as a function of mass have already been determined for the light fragment group and are shown in Figs. 10 and 11 respectively. The total mass yield of the light group shows a smooth increase up to the most probable mass of 108 amu and then a rapid decrease to symmetry, which shows a relatively high yield compared to  $^{235}\text{U}(n_{\text{th}},f)$ . The mean light and heavy masses are 106 amu and 140 amu respectively leading to an average emission of four neutrons. The mean kinetic energy as a function of the light fragment mass is in excellent agreement with the most recent PN1 data except for the very lightest masses below 90 amu, where there are discrepancies of about 1 MeV.

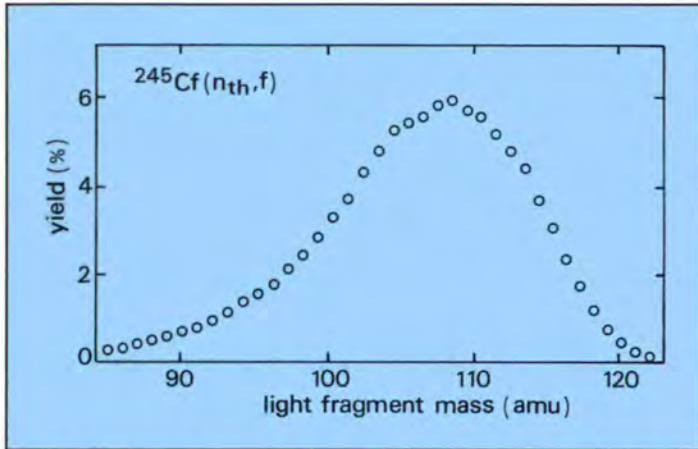


Figure 10:  $^{249}\text{Cf}(n_{th},f)$  mass distribution for the light fragment group after neutron evaporation.

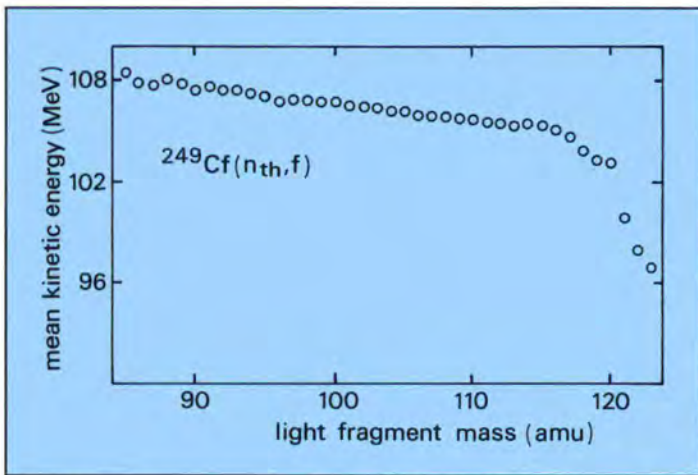


Figure 11: Mean kinetic energy as a function of the light fragment mass for the  $^{249}\text{Cf}(n_{th},f)$  reaction measured at PN8

Studies have also been carried out at PN8 of so called “hot” fragmentation in the  $^{235}\text{U}(n_{th},f)$  system and the data analysis is, due to its complexity, still underway.

### Fundamental Physics

The field of fundamental physics at the ILL covers research in many areas such as the testing of fundamental symmetries, the interaction of cold neutrons with bulk matter and, of course, the investigation of the basic properties of the neutron itself. For these experiments various neutron beams and instruments are used, both in the reactor (levels C and D) and in the neutron guide hall.

The new UCN-VCN source (TGV) on level D is operated in the framework of S-experiments. It was fully operational in 1987 giving a surprisingly stable flux. Installation of experiments is continuing with NESSIE, the gravity spectrometer, now partially installed and funding for the VCN-interferometer now available.

The VCN beam of the TGV (maximum intensity at 40 Å) has serviced several experiments. For 4 reactor cycles the superconducting magnetic hexapole storage ring (NESTOR-see last years annual report for details) from the University of Bonn was installed here. The ring served firstly for a neutron lifetime

( $\tau_\beta$ ) experiment where the number of surviving neutrons in the magnetic trap is recorded as a function of the trapping time  $t$  (up to one hour). This elegant method produces, based on a relative measurement, a value for  $\tau_\beta$  which can be interpreted as well as a lower bound. A preliminary result from some 50 hours of running is connected with an error of 16 sec. Secondly, an attempt was made to “weigh” the neutron using NESTOR as a “magnetic balance” (Fig. 12) with a sensitivity of  $10^{-25}\text{g}$ . For an absolute determination of the neutrons’ gravitational mass the vertical position of the neutron beam was measured as a function of the magnetic field strength. After stabilisation the beam is vertically scanned with a thin  $^3\text{He}$  detector. Fig. 13 shows the result of these measurements. The vertical position of the beam (for different apertures) is plotted versus the coil current which produces the magnetic force,  $\mu \cdot \delta B / \delta z$ , acting on the neutron. With the current detector system (as yet unoptimised) an absolute determination of the neutron gravitational mass is possible with an error of about 10 %.

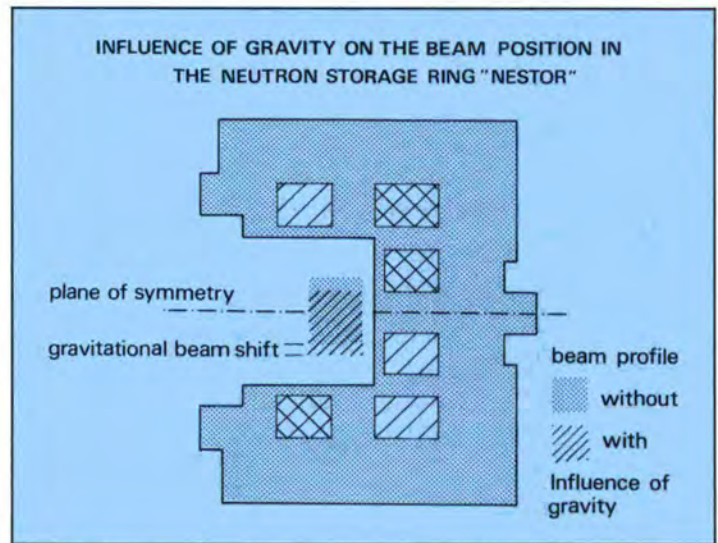


Figure 12: Influence of the gravitational potential on the beam position in the neutron storage ring NESTOR.

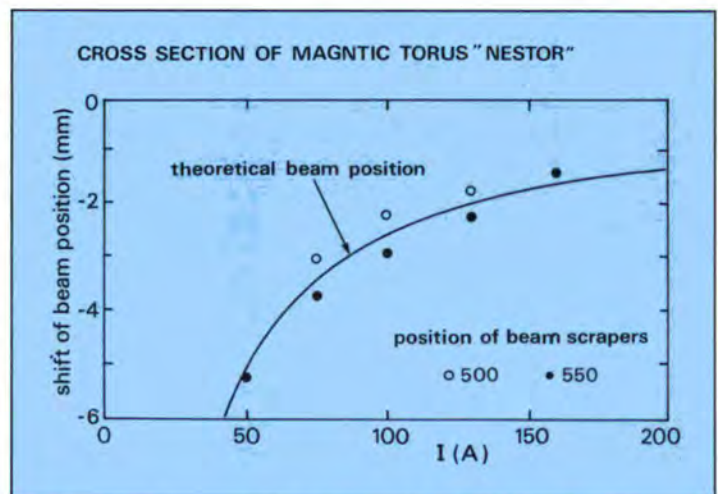


Figure 13: Vertical position of the beam versus the coil current producing the magnetic force  $\mu \frac{\delta B}{\delta z}$

A chopper was installed on the VCN beam to measure the  $^{235}\text{U}$  (n,f) cross section relative to that of the  $^6\text{Li}$ (n, $\alpha$ ) reaction at very low neutron energies. A preliminary analysis of the data taken so far shows no significant deviation from a  $1/v$  dependence between 1 and 100  $\mu\text{eV}$  which is in contradiction to theoretical predictions.

On the UCN beams of the TGV a variety of experimental setups have been employed during the past year. The neutron electric dipole moment (EDM) experiment has continued to take data with an essentially unchanged experimental arrangement - a preliminary analysis of which gives an uncertainty in the EDM of  $0.3 \times 10^{-27}$  em. However, the increased sensitivity resulting from the high flux means that systematic errors have become more apparent. Correction for these errors will, at worst, double the final uncertainty. Meanwhile work is underway to develop new magnetometers which will assist in the correction for systematic effects due to magnetic fields.

In another of the series of experiments attempting to measure the neutron  $\beta$ -decay lifetime UCN are stored in fluid walled bottles of identical height. By varying the bottle size UCN losses at the fluorated liquid wall coating can be eliminated. With a filling density of  $\sim 5$  UCN/ $\text{cm}^3$  in the velocity range 3-4.3 m/s and a vanishing background statistical errors of less than 1% are possible, - even with storage times up to one hour. The results from several runs are consistent. However, several sources of possible systematic errors have been, and are still, under investigation and no final result can be quoted until these are fully understood.

The neutron microscope was equipped with a new secondary mirror increasing the magnification to  $M = 243$ . With the unfortunate consequent loss in intensity the present system of recording the image of a slit which is slowly scanned through the neutron beam will have to be replaced with a position sensitive detector in order to record the whole image simultaneously.

The microscope was replaced by a very high resolution ( $< 1$  neV) neutron gravity diffractometer (Fig. 14) which exploits the vertical component of the neutron velocity. A horizontal beam of UCN is bent into a parabola by gravity and then reflected successively from a vertical-horizontal-vertical mirror system and focussed onto an exit slit. The position of the horizontal mirror determines the neutron fall height and thus the 'vertical energy component' (1 cm = 1 neV). Two experiments performed with this apparatus are summarised as follows:

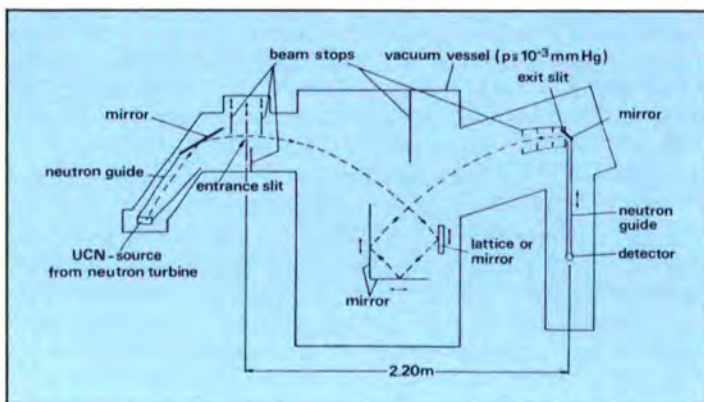


Figure 14: Schematic display of the neutron gravity diffractometer

a) UCN have been diffracted on a "blazed" grating with 1200 grooves/mm. From the linewidth of the +1st order, which is less than 1 neV, an estimate of the lower limit of the neutron coherence length of  $\xi > 10^6$   $\text{\AA}$  can be made.

b) Multilayer structures are seen by neutrons as a sequence of potential barriers and as such can give rise to quasi-bound neutron states. Fig. 15 shows the result of an experiment on a Cu-Al-Cu structure in which the resonance linewidth is about 3.6 neV leading to a bound state lifetime of 0.2  $\mu\text{s}$ . The Cu-Al-Cu-Al-Cu sequence forms a double potential well giving rise to a coupled neutron resonator and consequently the resonance line is split (by about 6.3 neV in this example).

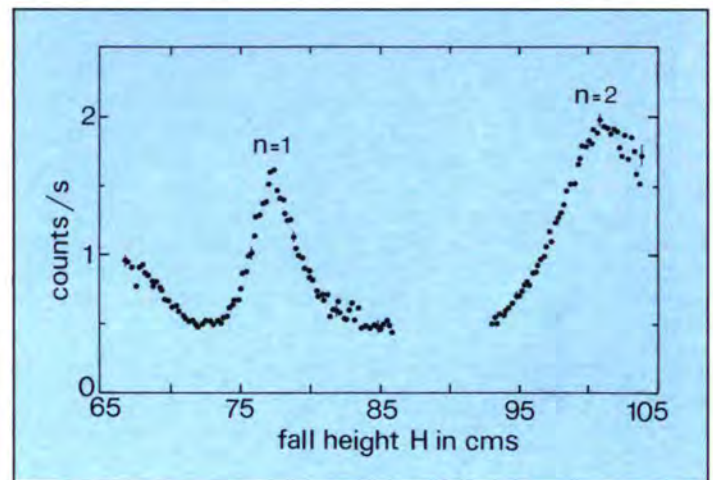


Figure 15: Neutron quasi bound states in a Cu-Al-Cu structure obtained with the gravity diffractometer

though most of the measurements mentioned above have already been performed at Garching they benefit greatly at the ILL from the 5000 fold increase in neutron flux available at the TGV.

SN5 has served mainly as a test facility for a variety of development work related to future improvements on the EDM experiment. To eliminate effects from spurious magnetic fields a magnetometer which monitors the flux in the same way as the UCN, over the whole storage volume, is under study. Mercury vapour ( $10^{-5}$  torr) seems to present the most promising solution. In relation to this various materials have been investigated to determine their reflection properties for both UCN and polarized Hg atoms. In addition D. Richardson (Sussex/ILL thesis student) has designed a UCN energy selector which will permit the future testing of the various properties as a function of UCN energy.

The experiment to measure the charge of the free neutron has completed taking data on the H18 beam position with a time-of-flight measurement of the neutron beam. The final result is  $q_n = (-0.6 \pm 1.1) \times 10^{-21}$  electron charges. Parts of this setup have been used by a Vienna, Garching, Bayreuth, ILL collaboration to investigate the inconsistency between the interference patterns produced by a 100  $\mu\text{m}$  slit using optical and neutron interference techniques.

Upscattering of UCN in a  $^4\text{He}$ -filled closed vessel has been directly observed for the first time in an experiment on the H17 beam. The deduced production rate and UCN density are in agreement with theory. This technique provides an opportunity to study interactions between excitations in the liquid at temperatures and wavelengths previously inaccessible.

The search for parity non-conservation (PNC) in radiative nuclear transitions was continued at the cold polarized neutron beam position SN7 and gave the first observation of PNC in resonance  $\gamma$ -ray scattering (Kurchatov, ITEP, Tübingen, ILL collaboration). The set-up for this experiment is shown schematically in Fig. 16. Circularly polarized  $\gamma$ -rays are produced in a target and irradiate a scatterer. For a suitable choice of the target and scatterer isotopes (i.e. recoil corrected energy of the  $\gamma$ -ray from the target close to an appropriate nuclear level energy of the scatterer) the polarized  $\gamma$ -rays are scattered resonantly. A helicity dependence in the scattering cross section would result in a different counting rate in the two BGO detectors and indicate PNC. Inversion of the neutron spin direction can be used to control the apparatus asymmetry. A sensitive experiment was performed using  $\text{Fe}(\bar{n},\gamma)$  as the source and Thallium as the scatterer for a 7.646 MeV  $\gamma$ -ray. A non-zero effect was detected for this combination with an asymmetry value of  $(4.8 \pm 1.5) \times 10^{-5}$ . This value is already corrected for a measured apparatus asymmetry of  $(2.3 \pm 1.6) \times 10^{-5}$ . A simultaneous measurement of the asymmetry in the direct gamma emission from the  $\text{Fe}(\bar{n},\gamma)$  reaction relative to the neutron spin direction gave a preliminary result of  $(-0.2 \pm 2.4) \times 10^{-5}$  which suggests that the observed PNC effect is indeed due to the resonance scattering in  $^{205}\text{Tl}$ .

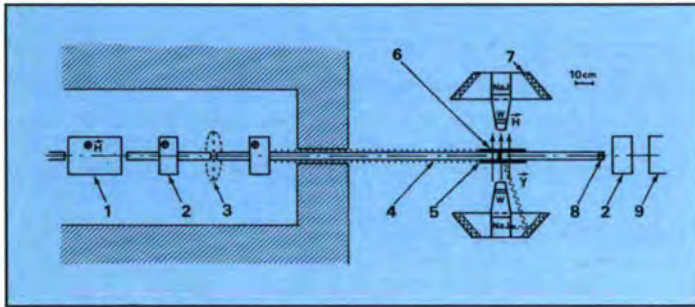


Figure 16 Experimental set-up of a resonance scattering experiment with polarized neutrons

- 1 supermirror polarizer
- 2 magnetic guide fields for neutron spin
- 3 current sheet spin flipper
- 4 copper neutron guide with solenoidal magnetic guide field
- 5 neutron shielding
- 6 target for  $(n,\gamma)$
- 7 scatterer
- 8 beam stop
- 9 polarization analyzer

For another neutron lifetime measurement a Helium filled drift chamber was placed at SN7 in a pulsed neutron beam (ILL, ISN, Univ. of Chambéry, LAPP collaboration). Neutron pulses of about 20 cm in length are produced (see technical section for details) and pass through the drift chamber (filled with a  $^4\text{He}$ - $^3\text{He}$ - $\text{CO}_2$  mixture) which works in a time projection mode in order to detect particle tracks. The electrons from the decaying neutrons and the protons from the  $^3\text{He}(n,p)$  reaction (which has a well known cross section) are measured simultaneously and are well discriminated events in the detector. The ratio of the decay electrons to the reaction protons, observed in a common time interval while the pulse is inside the chamber (0.6 ms) (Fig. 17), determines the neutron lifetime with no dependence on separate neutron flux or velocity measurements. A detailed evaluation of the data is underway.

## Special Beam Experiments

College 3 has three positions available at the end of the H22 neutron guide and these are used for a variety of experiments.

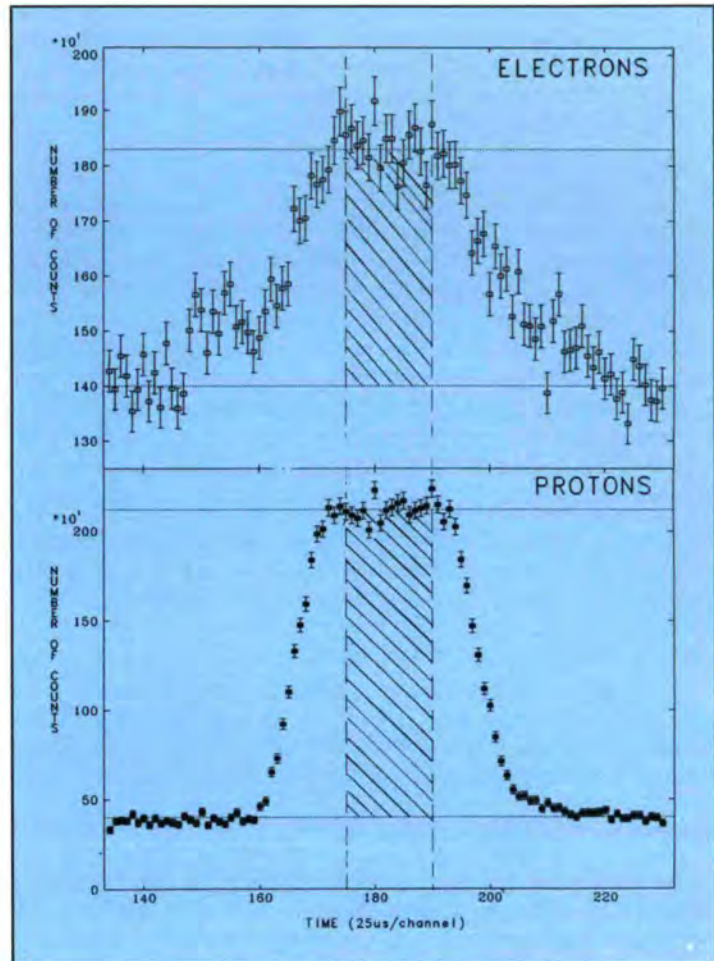


Figure 17: Event rates in the drift chamber as a function of time of flight of the neutron pulse. The ratio of the shadowed areas determines the neutron life time.

Various other beam positions throughout the ILL are also used for experiments coming under the College 3 umbrella.

A group from SCK/CEN Mol and CBNM Geel have continued their systematic studies of  $(n_{th},p)$  and  $(n_{th},\alpha)$  reactions at the H22D position. An investigation of the  $^{41}\text{Ca}(n_{th},\alpha)$  reaction has been completed and the  $^{138}\text{La}(n_{th},\alpha)$  and  $^{161}\text{Dy}(n_{th},\alpha)$  reactions have also been studied. An investigation of proton, deuteron and triton emission in the thermal neutron induced ternary fission of  $^{233}\text{U}$ ,  $^{235}\text{U}$  and  $^{239}\text{Pu}$  has been made. Data are currently being analysed.

At the H22E position an ILL-University of Grenoble collaboration continues its investigations into trace element concentrations in geological samples. A significant gain in sensitivity has been achieved by the installation of a dedicated sample chamber and a particular effort has gone into the further suppression of both scattered neutron and gamma background. This position was also used by a group from the University of Rouen to study the distribution of  $^6\text{Li}$ ,  $^{10}\text{B}$  and  $^{14}\text{N}$  in biological samples.

The H22F "end position" (S34) shares its beam allocation with the H22E position in order that neither position suffers from background contributions from the other. The primary use of the S34 position is as a gamma-gamma coincidence spectrometer for the study of nuclear structure. A group from the University of Sussex are regular users of this facility and recent studies include the  $^{199}\text{Hg}(n,\gamma)$  and  $^{187,189}\text{Os}(n,\gamma)$  reactions. Emphasis is placed on the identification of possible

IBM-2 mixed symmetry states. Of particular technical note was the use of pressurised gas targets in the study of Kr and Xe isotopes. This position was also used by a Belgrade-ILL-Sussex collaboration to investigate possible subnanosecond nuclear excited state lifetimes using the centroid shift method. Indications were found for measurable lifetimes of excited  $0^+$  states in  $^{106}\text{Pd}$  and  $^{184}\text{W}$  but further investigation is necessary.

A study of the neutron induced fission of the 26 min.  $^{235\text{m}}\text{U}$  isomer has been completed after experiments performed on the H12 and H14 beams (Collaboration with SCK/CEN Mol). The targets were prepared by collecting  $^{235\text{m}}\text{U}$  recoils produced by the  $\alpha$ -decay of  $^{239}\text{Pu}$  (Fig. 18). The cross section ratio of isomer to ground state was measured to be  $1.61 \pm 0.44$  and  $2.47 \pm 0.45$  for neutron spectra with peak energies of 5 meV and 28 meV respectively thus confirming that the neutron induced fission cross section of  $^{235\text{m}}\text{U}$  is larger than that of  $^{235\text{g}}\text{U}$ . This can be explained by the very different spin values ( $0^+, 1^+$  and  $3^-, 4^-$ , respectively) of both fissioning systems. The results also suggest the possible presence of a low energy resonance in the  $^{235\text{m}}\text{U}(n,f)$  cross section.

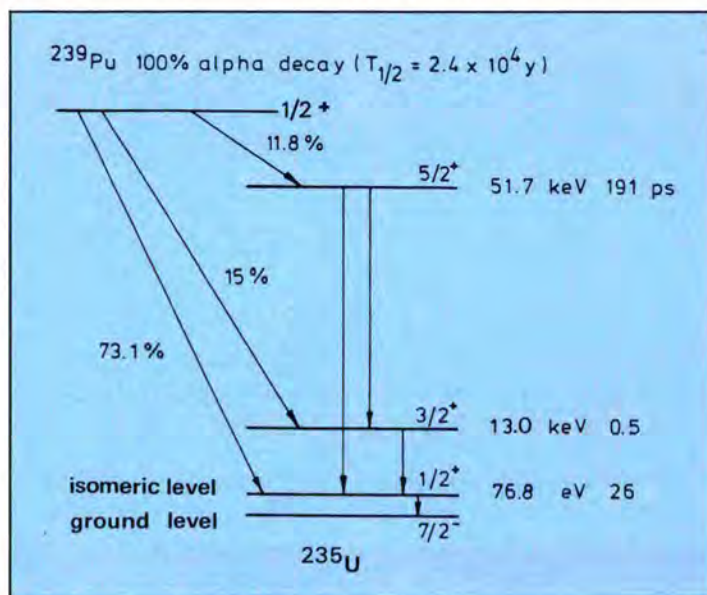


Figure 18: Population of the  $^{235}\text{U}$  lower levels due to  $^{239}\text{Pu}$   $\alpha$ -decay.

Finally a feasibility study of a new type of spin-echo spectrometer was also carried out on the polarised beam of SN7. In contrast to the conventional spin-echo method, the polarised neutrons are phase locked to localised high frequency fields and propagate in field zero. More details are given in the instrumental section of this report.

## Seminars

The college has continued with its seminar program dealing with such diverse topics as stellar astrophysics and the geology of the Himalayan mountains. Of particular note is the occasional series of seminars given by, and for, thesis students in order to better equip them for their future scientific careers.

Secretary: S. Robinson

### Astrophysics in College III

In recent years the instruments of College III were increasingly used to tackle problems in astrophysics. Astrophysics lives on scenarios and models. But these scenarios have to be supported by concrete physics data which come primarily from nuclear physics and which can in many cases be obtained by laboratory research.

Isotopic abundancies in the universe allow for very sensitive tests of astrophysics. Nuclei heavier than Iron 56 are produced in two different processes: In the first process, called the s-process, nuclei are produced by capture of slow neutrons, following a line which is always near the valley of nuclear stability. Crucial input values are neutron capture cross sections. However, as the s-process takes place at temperatures of about 30 keV, we also need to know the properties of the excited levels of the nuclei along and near the line of stability. The second process is the rapid neutron capture process. It takes place in explosive events. Input values to this process are the properties of very neutron rich nuclei far from stability.

Figure 19 shows the s-process paths in the region of the rare earth. Here  $^{176}\text{Lu}$  is a key nucleus which was recently investigated with the ILL nuclear physics spectrometers.  $^{176}\text{Lu}$  is interesting for astrophysics for two reasons: Its lifetime of  $3.6 \times 10^{10}$  years makes it suitable as astronomical clock; but this isotope may also be used as a sensitive thermometer for the s-process: it reveals an excited state with an excitation energy of 120 keV, which is populated by thermal excitation. It decays with a half-life of 3.7 h by beta emission to  $^{176}\text{Hf}$ . For these reasons  $^{176}\text{Lu}$  has been studied in detail at ILL. Many measurements relevant to the r-process have also been carried out at the Institut. Fig. 20 shows the very first part of the r-process starting at the "seed" nucleus  $^{56}\text{Fe}$ . The r-process is assumed to occur in cosmic explosions at temperatures of  $10^9$  K, at neutron densities of the order of  $10^{26}$  N/cm<sup>3</sup> and in times of the order of 1 to 100 seconds. The r-process path

proceeds very likely through the magic nickel isotopes until it reaches the closed neutron shell at  $^{78}\text{Ni}$ . Beyond this magic neutron shell the neutron binding energy drops to a very low level which, given the high temperatures of the r-process, will be populated in thermal equilibrium: In this case the (n, gamma) capture process occurs at the same rate as the inverse (gamma, n) process. Consequently, no further neutron can be captured to build heavier elements before nuclear beta decay sets in. Thus  $^{78}\text{Ni}$  provides a waiting position for the astrophysical r-process. Due to this mechanism the abundances of the heavier elements are largely determined by the lifetime of this doubly magic Nickel nucleus.

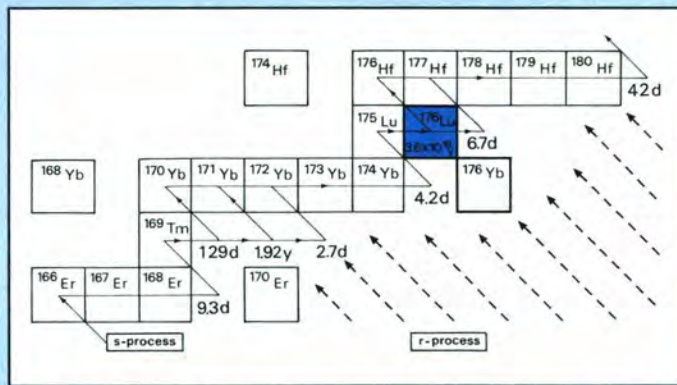


Figure 19: A section of the nuclear chart showing the path traced by the slow neutron capture nucleosynthesis process (s-process). Also shown is the  $\beta$ -decay feeding following rapid multiple neutron capture (r-process). It can be seen that  $^{176}\text{Lu}$  is shielded from r-process population by the  $\beta$ -stable nucleus  $^{176}\text{Yb}$  thus making it a suitable candidate for studying the characteristics of the s-process (see text).

The best way to understand the astrophysical processes is to produce the relevant nuclei in the laboratory. The neutron rich nickel isotopes from mass 70-74, and the neutron rich copper isotopes from mass 74-77 have very recently been identified on LOHENGRIN. They form a prominent part

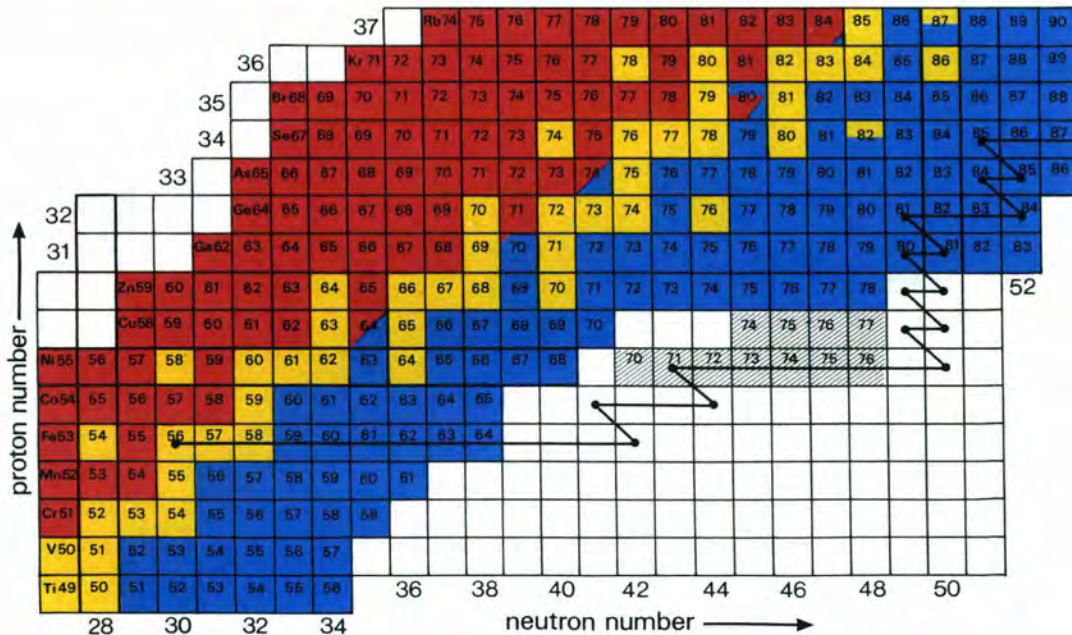


Figure 20: First part of the r-process paths, starting at the seed nucleus  $^{56}\text{Fe}$  (in black)

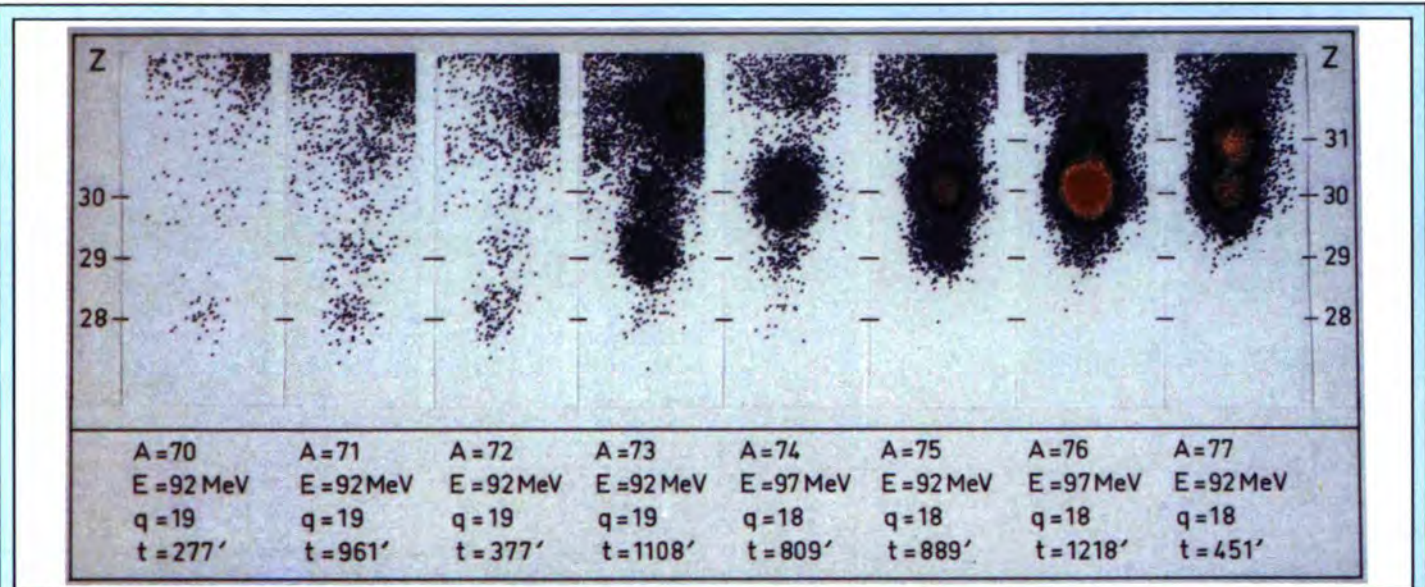


Figure 21: Identification of n-rich nuclei far from stability (at Lohengrin)

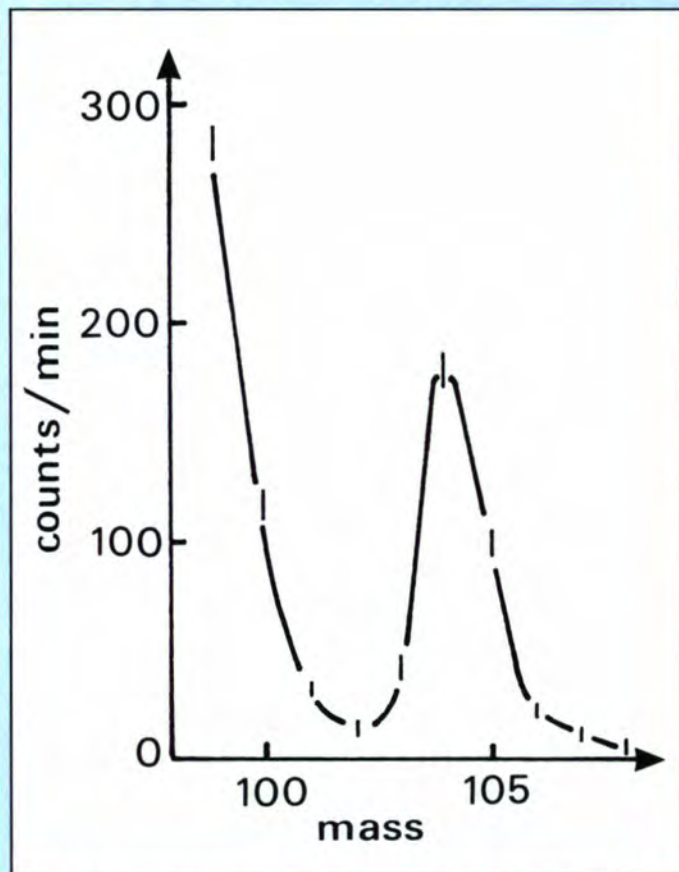


Figure 22: Beta-delayed neutron mass scan for  $A = 100$  to  $108$  at Lohengrin using a thick  $^{239}\text{Pu}$  target

of the r-process. The newest entry on our list is nickel 76, a nucleus having 12 neutrons in excess over the heaviest stable nickel isotope. At present the nuclear spectroscopy of these isotopes is in progress (Fig. 21). A further example of astrophysical application of nuclear data are measurements on delayed neutron emission from neutron rich nuclei. These neutron emission processes lead to

anomalies in the r-process abundance curve. Strong “delayed neutron” emitters have been observed on Lohengrin for masses 100, 104 and 105, as shown in Fig. 22. Delayed neutron emission tests also our ability to extrapolate nuclear properties into unknown regions using nuclear models. Furthermore they permit to derive neutron capture cross sections for unstable isotopes, which are inaccessible otherwise: the reason for it is that the decay mode of beta capture neutrons is just the inverse process to neutron capture.

Further measurements relevant to the r-process are shown in Fig. 23. Two neutron separation energies deduced from total energy release ( $Q_\beta$ ) measurements on LOHENGRIN and OSTIS are compared with recent mass formula predictions. On the neutron rich side, strong deviations occurred between measured and predicted values for “two neutron” separation energies. This underlines the importance of nuclear measurements for astrophysical applications.

H. Faust

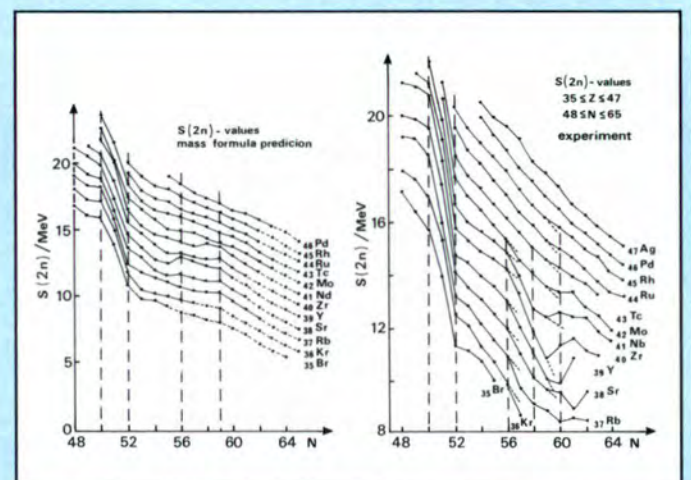


Figure 23: “Two neutron” separation energies deduced from  $Q_\beta$ -measurements on Lohengrin and Ostis.

# COLLEGE 4 STRUCTURAL AND MAGNETIC EXCITATIONS

## Members of the College

### I. Internal Members

M. Alba  
R. Currat  
L. Cussen  
D. Degenhardt  
B. Dorner  
J. Eckert  
B. Farago  
V. Frank  
P.H. Frings  
E. Gibbons  
H. Godfrin  
S. Hayden  
C. Lartigue  
H.J. Lauter  
A. Magerl  
J.L. Martinez  
A.P. Murani  
H. Mutka  
W. Petry  
B. Sarkissian  
O. Schaerpf  
H.P. Schildberg  
A. Severing  
W.G. Stirling  
C. Vettier  
C.M.E. Zeyen

### II. External Members

J.P. Boucher (CENG)  
J. Bouillot (Annecy)  
G. Dolino (U.S.M.G.  
St. Martin d'Hères)  
C. Filippini (LEPES, CNRS)  
P. Monceau (CRTBT, CNRS)  
L.P. Regnault (CENG)  
J. Rossat-Mignod (CENG)  
M. Vallade (U.S.M.G.  
St. Martin d'Hères)

## General Summary

The activity of College 4 members covers a wide field in solid state physics, centered around the dynamical properties of ordered structures and of magnetic systems.

Most of the experimental work is performed on three-axis spectrometers (IN1, IN3, IN8, IN12 and IN20) and on time-of-flight instruments (IN4, IN5, IN6 and D7). Over the years the emphasis has shifted progressively toward magnetic studies (magnetic excitations and magnetic phase transitions), which now amount to nearly 75% of all experimental proposals. This trend is at least partly due to the development of appropriate experimental sample environments and to the availability of polarised beam spectrometers (IN12, IN20, D7). It should continue in the coming years as the new cold-source three-axis spectrometer IN14 (with polarised beam option) becomes operational.

## Scientific Trends and Highlights in 1987

### Lattice Dynamics and Structural Phase Transitions

Several metallic systems have been investigated in which Fermi surface effects manifest themselves through elastic anomalies or structural instabilities. The b.c.c. form of titanium ( $\beta$ -Ti) is known to be stable above 883 C. In a series of experiments on IN3 and IN8 (1), using "in situ" grown single crystals the complete phonon dispersion of  $\beta$ -Ti has been measured at 1020 C. As shown in Fig. 24a, the dispersion curves exhibit two striking features:

- 1) The transverse  $[110]$  branch with  $[1\bar{1}0]$  polarisation is very low.
- 2) The longitudinal branch propagating in the  $[111]$  direction is soft near  $(2/3, 2/3, 2/3)$ , where scattered intensities are observed down to zero energy transfer (see Fig. 24b).

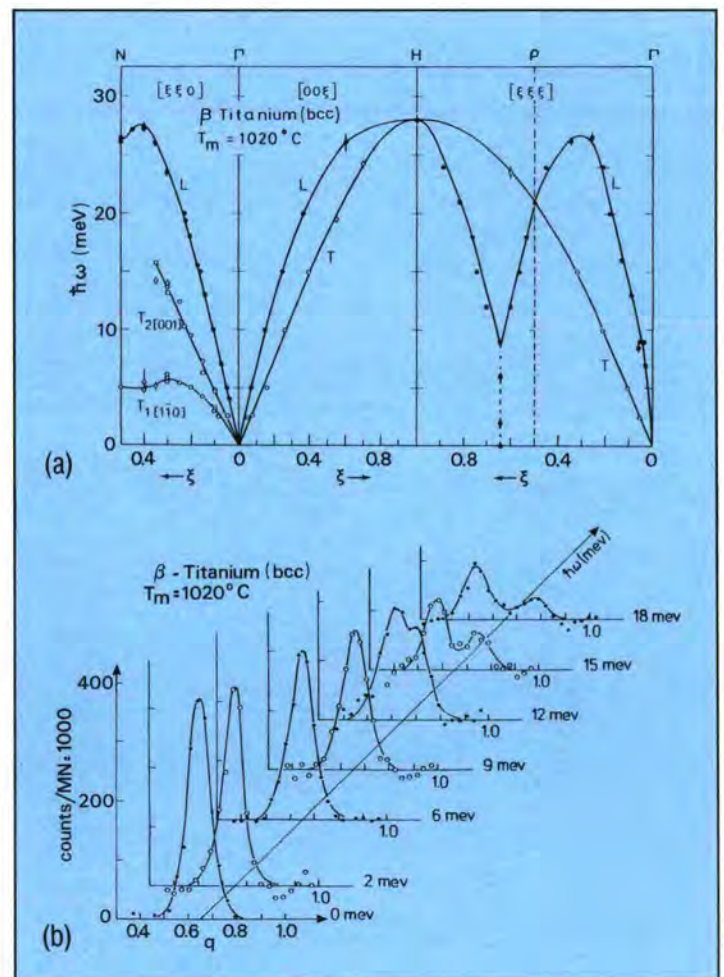


Figure 24: Phonon anomalies in b.c.c. titanium at 1020 C:  
(a) Dispersion curves;  
(b) Constant energy scans across  $q = 2/3(111)$ . Expt. 4-01-327.

The first observation is related to the occurrence of the b.c.c. to h.c.p. martensitic transformation which implies a low resistance to a  $\langle 110 \rangle [110]$  shear. The second observation corresponds to the tendency of the  $\beta$ -Ti lattice to form the so-called “ $\omega$ -phase”, which results from the displacement of two neighbouring (111) planes, while a third (111) plane stays at rest. It is believed that the anomalous self-diffusion rate and that of metal impurities, such as Fe, Co and Ni, is connected to the softness of the  $\beta$ -Ti lattice toward “ $\omega$ -phase”-type fluctuations, since the atomic displacements involved are parallel to the elementary diffusion jump vector.

Lattice-dynamical aspects of Peierls instabilities have been studied in a series of transition metal selenides [NbSe<sub>3</sub>, (TaSe<sub>4</sub>)<sub>2</sub>I, (NbSe<sub>4</sub>)<sub>3</sub>I, TaSe<sub>2</sub>, NbSe<sub>2</sub>], with one- or two-dimensional metallic properties (2). Recent IN12 results (3) have confirmed the dispersive character of the CDW-instability in 2H-NbSe<sub>2</sub>, in contrast to the incomplete mode-softening previously reported (4) in isomorphous 2H-TaSe<sub>2</sub>. The difference in microscopic behaviour for the two compounds can be correlated with observed differences in thermal and transport properties.

The physical relevance of a lattice-dynamical model is determined by its ability to reproduce measured phonon dispersion curves. When several models appear equally relevant, a direct test on the eigenvectors of the dynamical matrix is required. In practice this involves a comparison between calculated dynamical structure factors and measured integrated phonon intensities. In the case of Si, the intensity ratio between the LO and LA branches propagating in the [111] direction is determined by a single eigenvector phase factor for which several model predictions are available. The data shown in Fig. 25 (5) illustrate how phonon intensity measurements may be used as a sensitive tool to discriminate between various lattice-dynamical approaches.

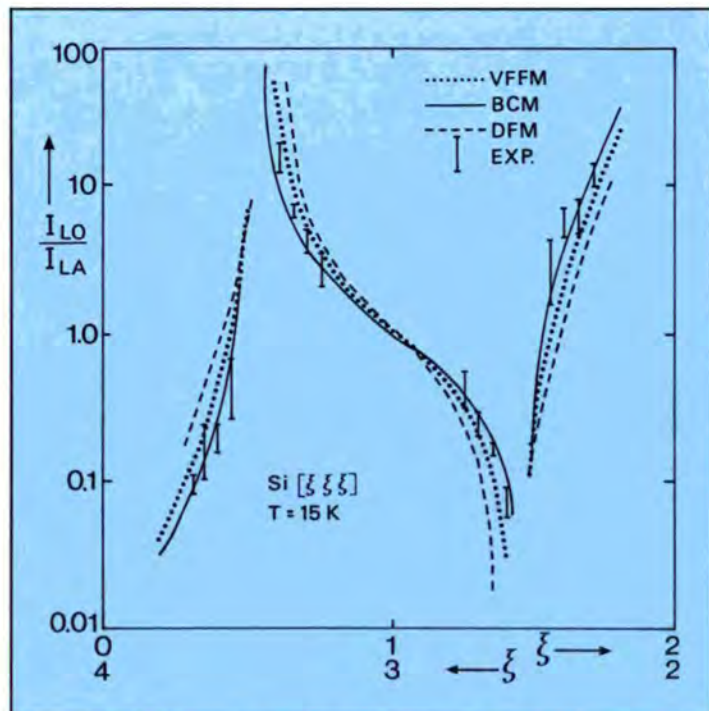


Figure 25: Measurement of the LO/LA intensity ratio in silicon as a function of momentum transfer along  $[\xi\xi\xi]$ . Experimental results (vertical bars) favor the bond charge model (solid line) over the valence-force field model (dotted line) or the dielectric function method (dashed line). Expt. 4-01-316 (IN8,  $k_1=4.1 \text{ \AA}^{-1}$ ,  $T=15 \text{ K}$ ).

The cubic antifluorite compound Li<sub>2</sub>O is of potential use as a Li-conducting solid electrolyte as well as a tritium-breeding blanket material in future fusion reactors. The characterisation of its lattice-vibrational spectrum and of the dynamic Li disorder at high temperatures is currently under way, using coherent and incoherent INS (6).

Crystalline acetanilide or ACN (C<sub>6</sub>H<sub>5</sub>NHCOCH<sub>3</sub>) is composed of chains of hydrogen-bonded ACN molecules with (–N–H...O=C–) distances comparable to those found in polypeptides and helical proteins. Recent optical work (7) suggests the existence of stable conformational defects (Davydov solitons), propagating without damping along the ACN chains. In biological systems, a similar mechanism has been proposed to account for the transport of energy in metabolic processes. So far, however, neutron results (8) only give indirect evidence, through acoustic anomalies, about the presence of such defects on the ACN chains.

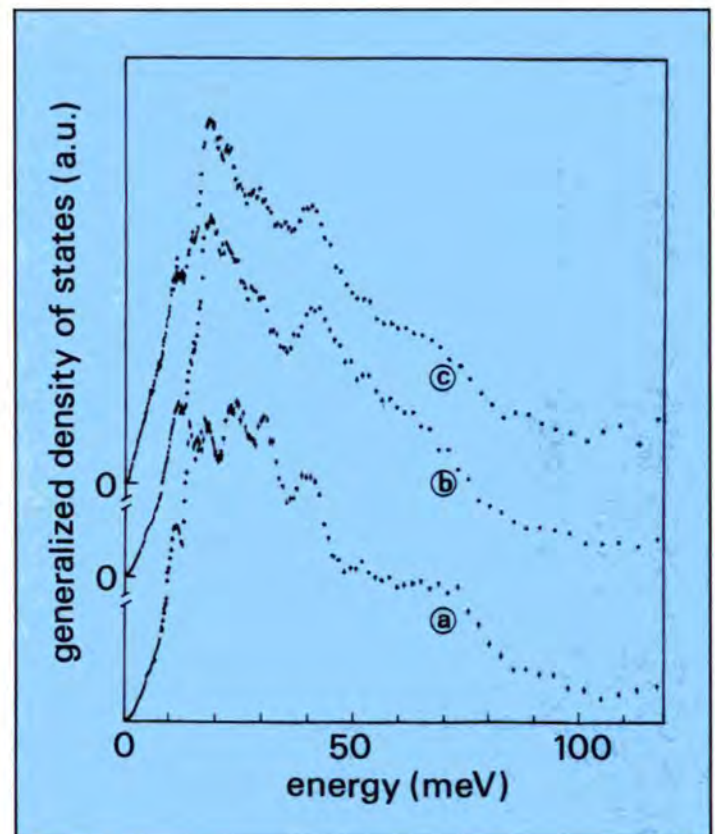


Figure 26: Generalized phonon density of states in YBa<sub>2</sub>Cu<sub>3</sub>O<sub>7-x</sub> at 295 K as obtained from IN6 TOF data (expt. 4-01-333): (a)  $x = .8$ ; (b)  $x = .0$ ; (c) YBa<sub>2</sub>(Co<sub>.45</sub>Cu<sub>2.55</sub>)O<sub>7</sub>.

A number of inelastic TOF measurements have been performed on powdered or polycrystalline samples of the new high-T<sub>c</sub> superconducting oxides (11). Although these compounds are predominantly coherent scatterers, it is possible to reconstruct the generalized phonon density of states, G(ν), from a suitable average of TOF spectra obtained over a range of detector angles (incoherent approximation). G(ν) curves measured at room temperature on IN6 are shown in Fig. 26, for several compounds in the Y-Ba-Cu-O family. Substantial differences are observed between the superconducting oxide ( $x = 0.0$ ) and the oxygen-deficient, non-superconducting compound ( $x = 0.8$ ), particularly in the range from 40 to 70 meV.

A large part of these differences, however, is likely to arise from differences in oxygen concentration (missing oxygen modes in the  $x = 0.8$  sample), rather than to phonon anomalies due to enhanced electron-phonon coupling in the superconducting compound. Note also the similarity between the  $G(v)$  curves from the superconducting and the Co-substituted samples, both having comparable oxygen concentrations but markedly different electronic properties (i.e. the Co-doped sample has tetragonal symmetry at room temperature, does not become superconducting and orders magnetically at 9K). Thus it seems that, in this class of materials the superconducting properties cannot be readily correlated to the presence of specific features in the phonon density of states. Further progress in this field will depend on the availability of single crystals of a suitable size for phonon dispersion curve measurements.

### Magnetism

The investigation of high energy magnetic excitations in 3d-transition metals continues, using the unique experimental capabilities of the hot source 3-axis spectrometer IN1. A series of such experiments, in the frequency range from 20 to 50 THz, have been performed, on hexagonal cobalt (10), chromium (11), and nickel above its Curie temperature (12). In cobalt, propagating spin waves could be observed with characteristic frequencies as high as 45 THz. In chromium, no magnetic signal was detected between 20 and 50 THz near magnetic zone centres, thus invalidating the previous identification of a 30 THz optical absorption peak with a direct interband electronic transition. However, magnetic inelastic scattering was observed away from the zone centres (e.g. at 20 THz). This new observation will be pursued in a forthcoming experiment.

At the other end of the frequency scale, the high resolution available on IN12 was found to be crucial in the determination of the spin wave spectrum of the antiferromagnetic garnet  $\text{Ca}_3\text{Fe}_2\text{Ge}_3\text{O}_{12}$  ( $T_N = 12.2\text{K}$ ). The magnetic structure of this compound is shown in Fig. 27a: it consists of two equivalent antiferromagnetically ordered  $\text{Fe}^{3+}$  sublattices, displaced by  $(1/4, 1/4, 1/4)$ . The interaction between the two sublattices is controlled by the nearest- neighbour exchange parameters  $J_1$  and  $J_1'$  and cancels out, in a mean field approximation. Neglecting the (very weak) magnetic anisotropy, a simple linear spin wave theory predicts two gapless acoustic magnon branches (corresponding to in-phase and out-of-phase spin precessions on the two sublattices), as well as optic magnon branches. The complete spin wave spectrum measured at 1.5K on IN12 (13) is given in Fig. 27b and shows good overall agreement with model calculations. Using tight instrumental conditions, a small gap ( $\sim 33\text{GHz}$ ) could be detected on the upper acoustic branch at  $q = 0$  (see Fig. 27c). This last feature, not predicted by linear spin wave theory is believed to arise from the interaction between magnetic fluctuations (thermal and quantum) on neighbouring  $\text{Fe}^{3+}$  sites, leading to a "dynamical" coupling between the two sublattices.

Cerium compounds are still the subject of intense investigation due to their unusual properties related to a coupling of the 4f electrons with valence or conduction bands. Depending on the strength of this coupling or hybridization, Ce ions can order magnetically or not.

As an example of weak hybridization, CeSb exhibits a complex magnetic behavior which is thought to arise from anisotropic mixing of the 4f electrons of Ce and the valence

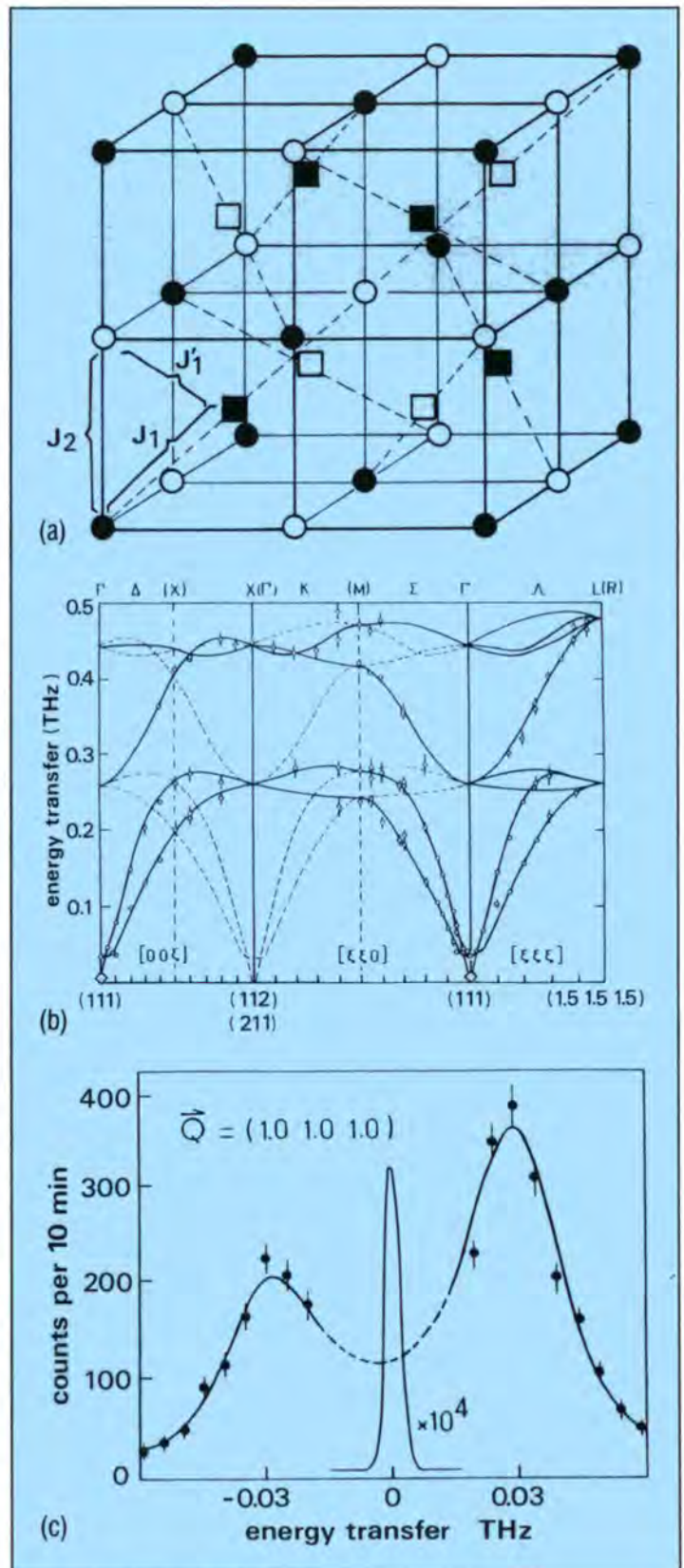


Figure 27: Spin waves in  $\text{Ca}_3\text{Fe}_2\text{Ge}_3\text{O}_{12}$ ; (a) The antiferromagnetic  $\text{Fe}^{3+}$  structure (up and down spin are shown as closed and open circles on one sublattice and by squares on the other); (b) Spin wave dispersion curves at 1.5 K (lines correspond to a model calculation with three exchange parameters); (c) Constant-Q scan showing the  $q = 0$  quantum gap at 1.5 K. The data are well fitted by a damped harmonic oscillator function including Bose occupation factor and varying analyser transmission (IN12,  $k_1 = 1.1 \text{ \AA}^{-1}$ ). Expt. 4-01-316.

bands. Earlier neutron scattering (14) and magnetization (15) measurements have shown the existence of a complicated magnetic phase diagram in which some phases would involve non-magnetic planes of Ce atoms stacked in between planes of fully magnetically ordered atoms. A recent experiment on IN20 (16) was performed in order to determine the nature of these planes by determining the nature of the magnetic excitations. In CeSb, due to the level scheme of Ce, excitations in the ordered planes are purely transverse, whereas magnetic excitations within planes of paramagnetic atoms have transverse and longitudinal character. Using the polarised neutron technique it was possible to discriminate between the two types of excitations and it was established that the non-magnetic planes in CeSb consist of paramagnetic  $Ce^{3+}$  ions in a  $\Gamma_7$  ground state. Furthermore excitations in ferromagnetic planes were found to be very sensitive to applied pressure (up to 30 kbar) which is reminiscent of the non-linear effects of the mixing with valence bands.

$CeCu_6$  is a typical heavy fermion system where the 4f-hybridization with the conduction bands leads to the appearance of Kondo-like fluctuations which prevent long range magnetic order. Such fluctuations have indeed been observed in  $CeCu_6$  (17). At low temperature,  $T \sim T_k$ , anisotropic magnetic correlations between fluctuating moments have been observed at incommensurate positions as well as at zone boundary points in reciprocal space (18). This indicates the existence of both single-site and intersite spin fluctuations. Furthermore the correlation lengths are found to saturate at temperatures below  $T_k/2$ , consistent with the absence of long range magnetic order. Application of a magnetic field destroys these antiferromagnetic correlations; under high magnetic fields, the Ce ground state doublet is Zeeman-split and propagating excitations within this state have been found. The low field measurements have confirmed the existence of a gap in the magnetic response, in agreement with some theoretical predictions (19).

Work on the magnetic excitation spectra of actinide materials has continued with experiments on IN1 (USb) and IN8 (UTe, NpSb). In the first of these, the temperature dependence of the upper branch ( $E \sim 7$ THz) of antiferromagnetic USb was determined for the first time (20); a search for a predicted higher energy crystal-field excitation was inconclusive. Studies on the ferromagnet UTe have continued with measurements on a sample cooled in a magnetic field to produce a monodomain (21). For wavevectors parallel to the moment direction ([111]), the spin wave branch has been followed to the zone boundary while the mode is damped out rapidly in the direction perpendicular to the moment, as shown in Fig. 28. This anisotropy in the spin-wave dispersion is very unusual in an actinide compound. Unfortunately, despite extensive surveys around the antiferromagnetic zone centre, only tentative evidence for spin-wave scattering could be obtained in NpSb (22). This result was unexpected as, in many respects, neptunium antimonide resembles uranium antimonide. Nevertheless strong critical scattering was observed and was found to exhibit an extremely large correlation-length anisotropy, almost identical to that found in USb and much larger than in PuSb.

Polarisation analysis can be used to separate nuclear coherent, nuclear (spin-)incoherent and magnetic scattering. To decide whether magnetic fluctuations are present in the high temperature superconductor  $YBa_2Cu_3O_7$ , measurements were performed (23) on the instrument D7 used in the integral mode (no energy analysis), but with polarisation analysis. The total magnetic intensity, within the accessible energy window, was found to be vanishingly small. This negative result is of

considerable significance in the context of current speculations on the mechanism for superconductivity in this family of compounds.

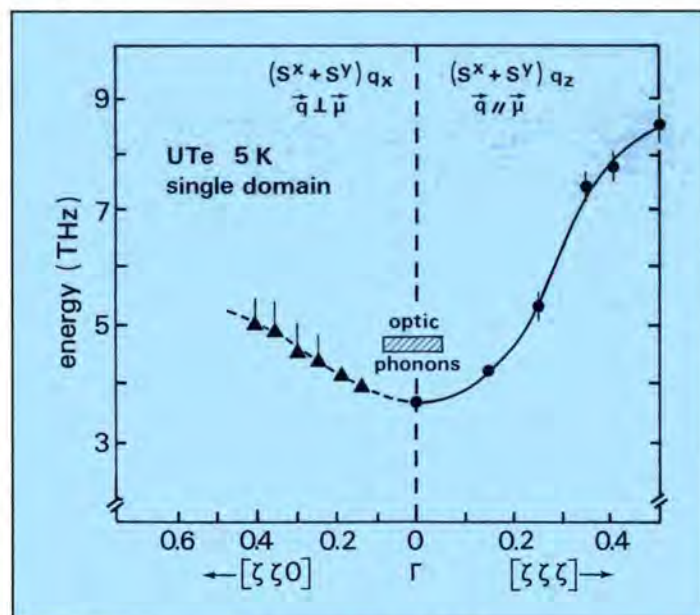


Figure 28: Dispersion curve for monodomain UTe, with magnetic moment parallel to [111]. Expt. 4-03-446 (IN8).

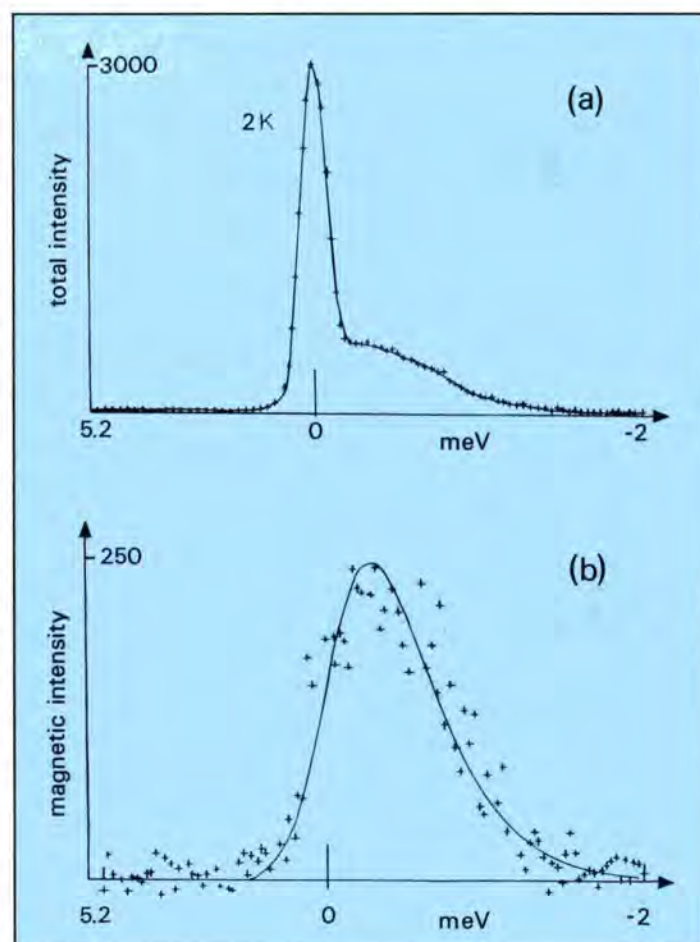


Figure 29: Separation between magnetic and incoherent scattering in  $Na_3Cr_2(PO_4)_3$  at 2 K: (a) magnetic and incoherent scattering; (b) magnetic scattering only. The quasielastic magnetic scattering is fitted to a Gaussian deformed by the factor  $\omega^2$  Bose factor. Expt. 5-33-221 (D7).

In the superionic conductor NASICON ( $\text{Na}_3\text{Cr}_2(\text{PO}_4)_3$ ), short range antiferromagnetic Cr-Cr correlations give rise to quasielastic scattering which overlaps with the incoherent scattering cross-section due to the dynamically disordered sodium ions. Fig 29a shows the spin-flip cross-section obtained using D7 in the polarised TOF mode (24), with incident spin polarisation along the vertical direction ( $\sigma_z \uparrow \downarrow$ ). Fig 29b shows the magnetic cross-section alone, as obtained from the combination:

$$\sigma_x \uparrow \downarrow + \sigma_y \uparrow \downarrow - 2*\sigma_z \uparrow \downarrow$$

At  $T=2\text{K}$ , the magnetic intensity is purely quasielastic, while the incoherent intensity which is obtained from the combination:

$$\sigma_x \uparrow \downarrow + \sigma_y \uparrow \downarrow - 3*\sigma_z \uparrow \downarrow$$

appears as purely elastic (static disorder).

Further measurements are planned at higher temperatures, when both components are expected to have comparable quasielastic linewidths.

The neutron scattering from the amorphous ferromagnet  $\text{Fe}_{83}\text{B}_{17}$ , has been studied using IN20 with full polarisation analysis (25). The elastic scattering shows that in the ferromagnetic state the spins are not wholly aligned along an applied field. The inelastic scattering shows that there are low energy excitations in addition to the long wavelength spin waves. This finding may be correlated to the observed difference in spin wave stiffness as deduced from neutron scattering and magnetisation measurements.

## Special Beam Time on IN3

During 1987, 55 days of special beam time were made available to the British group (61 days in 1986), and 44 days to the German group (see report in box, Coll. 9).

This year saw the replacement of all motor control modules and Tanzboden motors and of the monochromator. Despite some teething problems, which resulted in some lost beam time, these changes appear to have improved the performance of the instrument significantly.

In Jan. 87, inelastic measurements were performed on a variety of systems, such as the important solar cell material  $\text{CuInSe}_2$ , the magnetic perovskite  $\text{NdMnO}_3$ , the ternary magnetic superconductor  $\text{HoRhSn}$  and the binary alloy  $\text{Fe}_{30}\text{Cr}_{70}$ . This last work, which showed much less magnetic influence on the phonon frequencies than expected, has now been completely analysed and will be published soon.

Following recent predictions of anomalous spin waves in multiple-Q magnetic structures, a study on a MnNi alloy showed evidence for a dramatic changes in the inelastic magnetic scattering associated with phase transitions between the various possible ordered structures. This work will continue in the coming year.

The origin of critical scattering in  $\text{KMnF}_3$  was investigated. The scattering which is observed well above the structural phase transition temperature, has been attributed to defect scattering. However, measurements on crystals with an increased defect concentration ( $\text{KMn}_{1-x}\text{Mg}_x\text{F}_3$ ) did not show any enhancement of the effect.

Finally, the exciton spectrum of a PrEr alloy was investigated. The four low energy branches were completely mapped except

for a small wavevector region where the IN3 resolution was insufficient to separate the branches. This work will be continued next year on IN12.

Secretary: R. Currat

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- (2) Expt. 4-01-307 and 4-01-308; P. Monceau, L. Bernard, R. Currat, F. Levy and J. Rouxel.
- (3) Expt. 4-02-232; C. Ayache, P. Molinie and R. Currat.
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- (9) Expt. 4-01-331; M.L. Vrtis, G.W. Crabtree, W.G. Stirling and A.P. Murani. Expt. 4-01-332; B. Renker, F. Gompf, P. Schweiss, H. Rietschel, E. Gehring and, N. Nucker. Expt. 4-01-333; P. Monceau, R. Currat, A.J. Dianoux, P. Strobel, J.G. Bednorz and J.L. Tholence. Expt. 4-01-335; P. Day and K. Prassides.
- (10) Expt. 4-03-434; G.L. Squires, T.G. Perring, K.R.A. Ziebeck, W. Prandl, A.M.B.G. de Vallera and S.M. Hayden.
- (11) Expt. 4-03-433; P.W. Mitchell, J.G. Booth, D. Mc Paul and W.G. Stirling.
- (12) Expt. 4-04-163; D. Mc Paul, H. Mook, W.G. Stirling and S.M. Hayden.
- (13) Expt. 4-03-454; Th. Brueckel, B. Dorner, A.G. Gukasov, V.P. Plakhty, W. Prandl, E.F. Shender and O.P. Smirnov.
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- (17) Expt. 4-03-453; J. Rossat-Mignod, J. Flouquet, L.P. Regnault and C. Vettier.
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- (25) Expt. 4-05-188; R.A. Cowley, L. Cussen and N. Cowlam.

## Measurements of phonons by inelastic X-ray scattering

With the new synchrotron radiation sources intense X-ray beams become available. These provide the possibility of developing inelastic X-ray scattering as a complementary technique to inelastic neutron scattering (for more details about the complementarity of neutrons and X-rays, see annual report 1985, p. 31). A group of scientists around J. Peisl at the Ludwig Maximilians Universität München with the cooperation of B. Dorner, ILL constructed the instrument "INELAX" /1/ on the storage ring DORIS at HASYLAB, Hamburg, see Fig. 30.

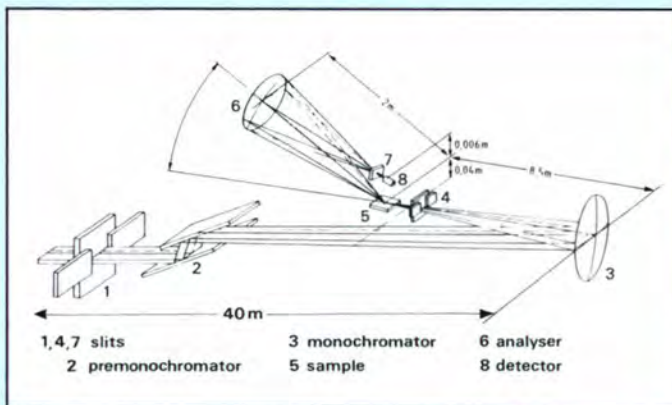


Figure 30: The instrument INELAX

To obtain a resolution  $\Delta E/E$  of about  $10^{-6}$ , the monochromator III and the analyser VI have to operate at Bragg angles very near to  $90^\circ$  (backscattering) and high order reflections from perfect crystals (Si(777)) have to be used. These crystals are spherically curved for focussing to get sufficient intensity onto the sample and finally into the detector. The diaphragms IV and VII define the geometrical part of the resolution. The premonochromator, unsymmetrically cut Ge(220), is necessary to take the heat load due to the strong synchrotron radiation off the monochromator.

Inelastic scattering has been performed on pyrolytic graphite observing a high energy maximum in the density of states and on a single crystal of h.c.p. Beryllium to measure phonons /1,2/. Fig. 31 shows the dispersion curve for longitudinal modes along the  $[00\xi]$  direction in Be as measured with inelastic X-ray scattering. For comparison inelastic neutron scattering data /3/ are given as well. This particular direction has the advantage that only the longitudinal modes are visible and that the acoustic modes go over continuously into the optic ones.

These results demonstrate the feasibility of inelastic X-ray scattering with a resolution of about 50 meV (to be improved). The average count rate was about 5 photons per min. The instrument is now moved to the hard X-ray wiggler beam at HASYLAB, where an intensity gain of a factor 10 is expected. A further gain will be obtained when the instrument will be installed on the European Synchrotron Radiation Facility.

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 surfaces; electronic transitions; etc.

B. Dorner

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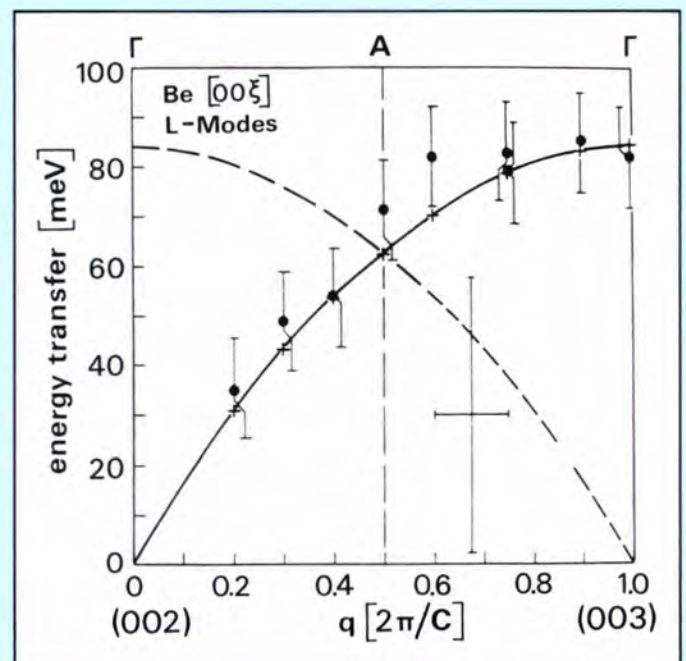


Figure 31: Phonon dispersion curve in Be. • phonons observed with inelastic X-ray scattering /2/. A indicates the Brillouin zone boundary and the dashed curve the invisible modes.

## Phonons in the commensurate phase of D<sub>2</sub> on graphite

by, H.J. Lauter, V.L.P. Frank.  
see Box in the section of College 9.

# COLLEGE 5 CRYSTAL AND MAGNETIC STRUCTURES

## Members of the College

### 1. ILL Members

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## GENERAL SUMMARY

During 1987 many of the instruments used by the college functioned routinely, and on these full research programmes were carried out. The exception is D3B, which was installed on the D5 position during the second half of the year. Alignment and test are now nearly completed, and the first measurements should start early 1988. The instrument D1A is now almost completely assigned to "special beam" use, and there is already a long queue of demands ranging from basic research to engineering applications.

The wave of activities following the discovery of the new high- $T_c$  superconductors hit the college in March, and soon several members were deeply engaged in research on these exciting compounds. The most used technique was powder diffraction, but also single crystal work was carried out. To ensure that samples were measured as soon as possible the quick access procedure foreseen for such cases was employed. This procedure is rarely used, but worked well in the present case, partly because it was possible to transfer part of the studies onto the special beam instrument D1A, thus reducing the perturbations on the normal schedule. Research activity in this area remains high, as reflected in the large number of proposals for the autumn session of the scientific council, but now most of the work can be carried out by normal scheduling.

## SCIENTIFIC HIGHLIGHTS IN 1987

### Crystallography on non-magnetic systems

#### High $T_c$ superconductors, powder diffraction

As indicated above the great excitement of the year was the discovery of high temperature superconductivity by Bednorz and Müller, and the efforts to solve and understand the structures of these materials. Neutron powder diffraction proved a unique tool for this purpose. The structure of the "100K" superconductor  $\text{YBa}_2\text{Cu}_3\text{O}_7$  was obtained at several temperatures between 5K and 300K (collaboration with groups from Grenoble and Caen), and confirmed by the simultaneous room temperature work at Argonne and RAL. Earlier X-ray single crystal measurements had found disorder of oxygen defects in a perovskite superstructure, but the neutron powder work showed that all oxygen "defects" were ordered. The same copper oxide planes were present as in the original "35K" Bednorz and Müller material  $\text{La}_2\text{CuO}_4$ , but an exciting new feature was observed, 1D copper oxide chains.

The measurements showed that there was some transverse disorder of oxygen O4 within these chains. Even at 5K, chain oxygens move toward adjacent channels through which oxygen conduction occurs at high temperature. Further studies, using D2B up to 1000K, showed how this disorder increased (fig. 32), with O4 oxygen being lost to obtain finally a well ordered tetragonal form:  $\text{YBa}_2\text{Cu}_3\text{O}_6$ . The possibility of coupling between these vibrations associated with oxygen conduction, and valence fluctuations of copper within the chains, was suggested.

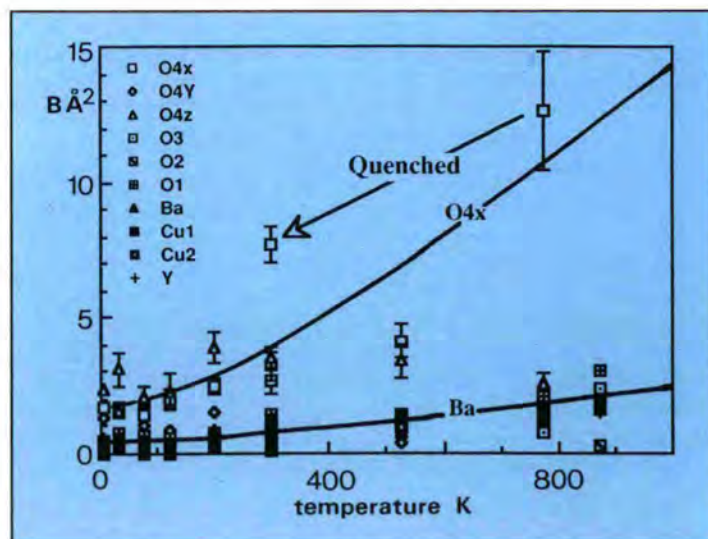


Figure 32: Debye-Waller factors expressed as  $\exp. (-B \sin^2 \theta / \lambda^2)$  as a function of temperature.

These structural measurements were repeated at many different temperatures with short wavelengths (1.05 Å) on D2B to obtain the structure to high resolution (collaboration between groups from University of Geneva, Würenlingen, CNRS Grenoble and ILL). In particular, the temperature dependence of the atomic vibration was examined carefully. These demonstrated very clearly that the "soft mode" involved essen-

tially transverse vibration of O4 across the copper oxide chain. The zero point motion of all atoms is normal, except for O4, where it is particularly large in this direction. This suggested that O4 may be moving in a double potential well, with minima on either side of the chain. A further search for ordering on samples annealed for several days at 240 K, revealed no superlattice reflections. O4 ordering within the chain may be rather short range ( $< 100 \text{ \AA}$ ).

Several very pure samples were examined; the best showed essentially complete O4 occupation, zero O5 occupation within the channels, and no impurity lines at all. Many other samples from other groups were also characterised for purity, oxygen stoichiometry etc.

In particular the sample used to search for magnetic fluctuations in  $\text{YBa}_2\text{Cu}_3\text{O}_7$  was carefully characterised. This important experiment (University of Tübingen and ILL) showed decisively the absence of magnetism in this material, at least for fluctuations within large limits of momentum and energy transfer.

Of course, a careful search for magnetic ordering down to 1.5K was also made with powders on D1A and D2B. No such ordering in pure stoichiometric superconducting  $\text{YBa}_2\text{Cu}_3\text{O}_7$  was found. However, in a very nice D1A experiment on an Oak Ridge sample of  $\text{GdBa}_2\text{Cu}_3\text{O}_7$  98% enriched in  $\text{Gd}^{160}$  to reduce absorption, 3D magnetic ordering of Gd moments below 2.2 K (Fig. 33) was demonstrated (Warwick, Oak Ridge and ILL). No ordered Cu moments were obtained, but it remains unclear how the Gd moments can be coupled across the almost  $12 \text{ \AA}$  c-axis spacing.

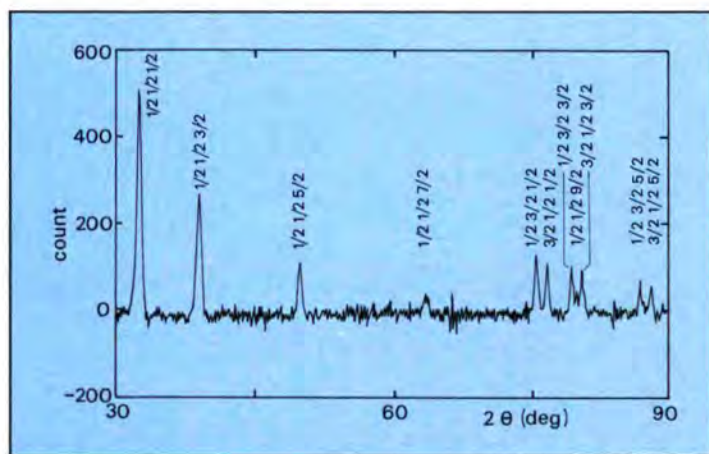


Figure 33: Magnetic scattering for  $\text{GdBa}_2\text{Cu}_3\text{O}_7$  at 2.2 K. Powder data from D1A.

These experiments to obtain very precise structures and vibrational amplitudes, and to investigate magnetic ordering with large d-spacings, are probably best done on reactor based machines where high resolution can be obtained both at small scattering angles (for magnetism) and at short wavelengths (for vibrational amplitudes). The superconductors are good examples of real materials, where the experimental resolution is ultimately limited by the particle size of the sample, in this case the domain structure.

### High $T_c$ superconductors; single crystals

The first publications to come from the new instrument D19 were also concerned with the high  $T_c$  superconductors, and the study was a collaboration between groups from the University of Paris and ILL. In this case single crystals were used,

and as the superconducting phase was twinned, the 2-dimensional position sensitive detector proved to be very useful. Both the tetragonal non-conducting phase ( $\text{YBa}_2\text{Cu}_3\text{O}_{6.2}$ ) and the orthorhombic, conducting phase ( $\text{YBa}_2\text{Cu}_3\text{O}_{6.7}$ ) were studied at several temperatures.

For the tetragonal phase the increase in oxygen vacancies compared to  $\text{YBaCu}_3\text{O}_7$  is confined to the sites in the plane midway between pairs of closest barium atoms, and no significant differences were observed between the structures at 20K and 298K. For the orthorhombic case a special effort was made to study the quadruply split lattice point due to the interchange of the orthorhombic a and b axis about the (110) planes. This is shown schematically in figure 34, and figure 35 depicts the resulting diffraction pattern. The intensity ratios of the individual reflections of some split peaks are shown to be particularly sensitive to the relative occupations of the oxygen sites in the plane between pairs of closest Ba atoms, and confirm that the oxygen lies on just one site. Particularly large thermal parameters were observed for Cu1, O1 and O4. The sample was studied at 40 and 100K, but unfortunately the temperature dependent data were not precise enough to distinguish between static and dynamic disorder. This is probably mainly due to the small size of the crystal ( $\sim 0.1 \text{ mm}^3$ ). Recently, larger crystals have been obtained and have been tested on D9, and it is hoped that in 1988 these studies can be pursued successfully.

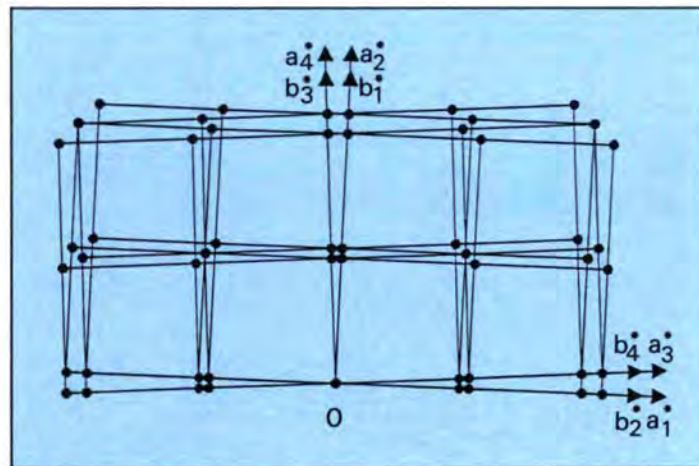


Figure 34: a-b plane of  $\text{YBa}_2\text{Cu}_3\text{O}_7$  showing the twinning pattern.

### Quasi-crystals

Quasi-crystals have been the focus of great interest since their discovery. They have been first identified in rapidly solidified alloys and later in conventionally cast Al-Li-Cu alloys as the result of a solid state nucleation and growth process. Millimeter size triacontahedral grains (fig. 36) were easily identified and isolated for detailed X-ray measurements. Dendrites were observed to grow along common 5-fold axes of the triacontahedral. Such a centimeter size piece of dendrite, with a 5-fold symmetry pyramidal shape, was produced and isolated for transmission Laue and four-circles neutron diffraction measurements (D10). Misorientation of the triacontahedra within the pyramid happens to be small enough to allow real "single-crystal" data to be collected. The major reflections observed only exhibited 2-fold, 3-fold or 5-fold point symmetries. Furthermore, the measured angles between these directions were consistent with an icosahedral structure i.e.  $63.43^\circ$  between 5-fold,  $31.72^\circ$  between 2-fold and 5-fold,  $37.37^\circ$  between 5-fold and 3-fold axes (within experimental resolution).

The peaks observed in the single crystal diffraction scans were found to be mostly indexable with six indices that belong to a primitive icosahedral Bravais quasi-lattice. Example of diffraction scans, with the scattering vectors along two different sort of 3-fold axes are shown in figure 37, with peak indexation and question marks for unindexable peaks. The experiment allowed to specify unambiguously the point group symmetry to be  $m\bar{3}5$ .

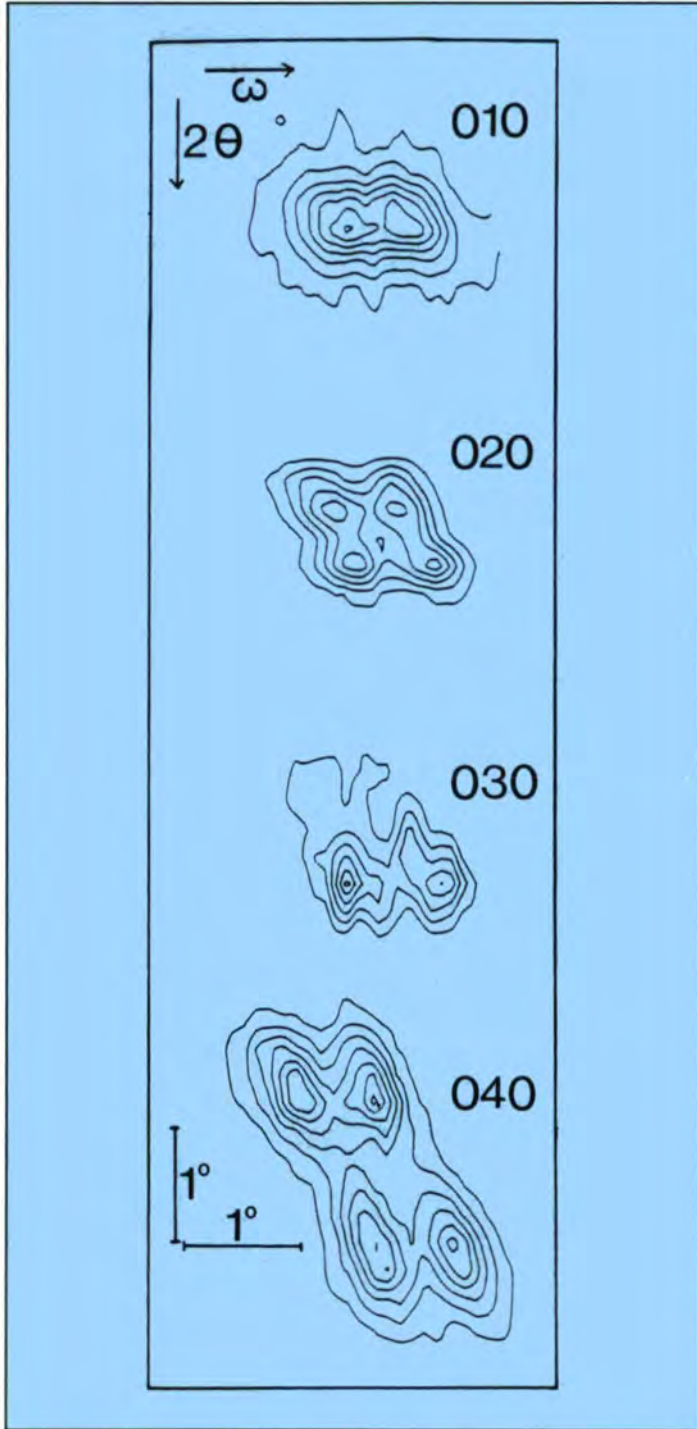


Figure 35: Recording for  $YBa_2Cu_3O_7$  on a 2-dimensional detector of the split peaks (OKO) due to twinning.



Figure 36: Millimeter size grain which is a single quasicrystal of  $Al_6Li_3Cu$  (courtesy of Pechiney, Centre de Recherche, Voreppe)

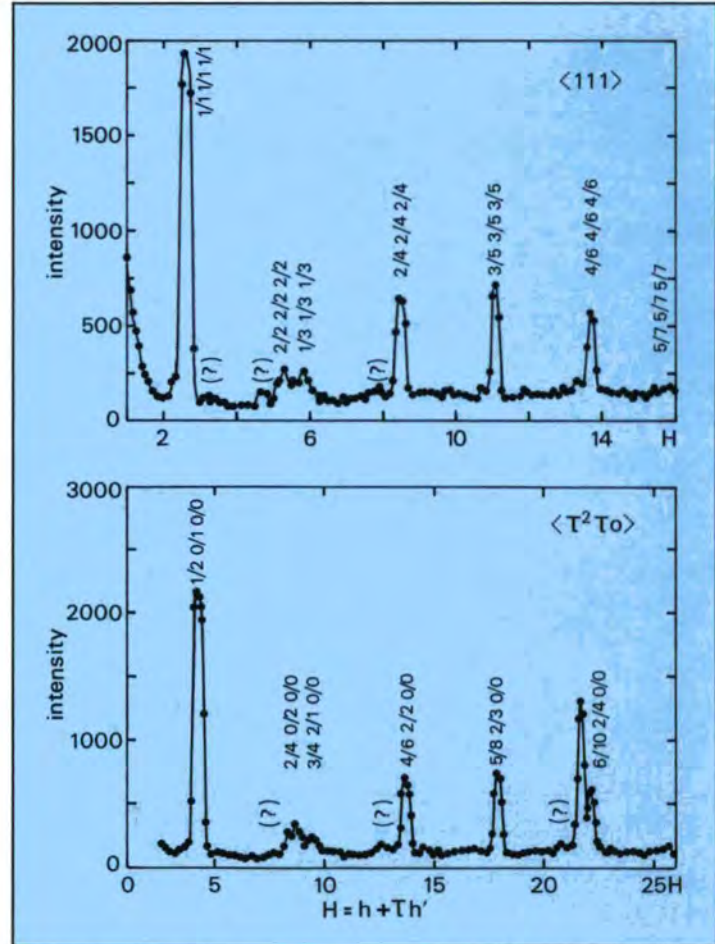


Figure 37: Diffracton scans along two different sorts of 3-fold axes in a quasicrystal of  $Al_6Li_3Cu$ .

### Scattering patterns observed with a PSD

The 2-dimensional Position Sensitive Detector on D19 served on several occasions in the discovery and detailed analysis of non-Bragg scattering. One example was the study of an Al-Li-Cu based structure  $Al_6(Cu,Zn)Li_3$ , which is tetragonal with  $a = 14 \text{ \AA}$  and  $c = 82 \text{ \AA}$  (collaboration with Pechiney Research).

In the first instance D19 permitted the collection of a large number of reflections, and as the long c-axis was mounted along the long axis of the detector, which is  $\pm 32^\circ$  in the vertical direction, several reflections could be recorded simultaneously. During these measurements additional large intensities were observed between some Bragg reflections. An example is shown in figure 38, where a scan through the 641 is depicted. Additional scattering is detected between 640 and 641, but a similar effect is not found between 641 and 640. Employing the complete information the Bragg intensity could be separated from the additional scattering. In the present case this scattering is probably not due to any characterisable disorder in the crystal, and the structure analysis is now under way on the Bragg intensities.

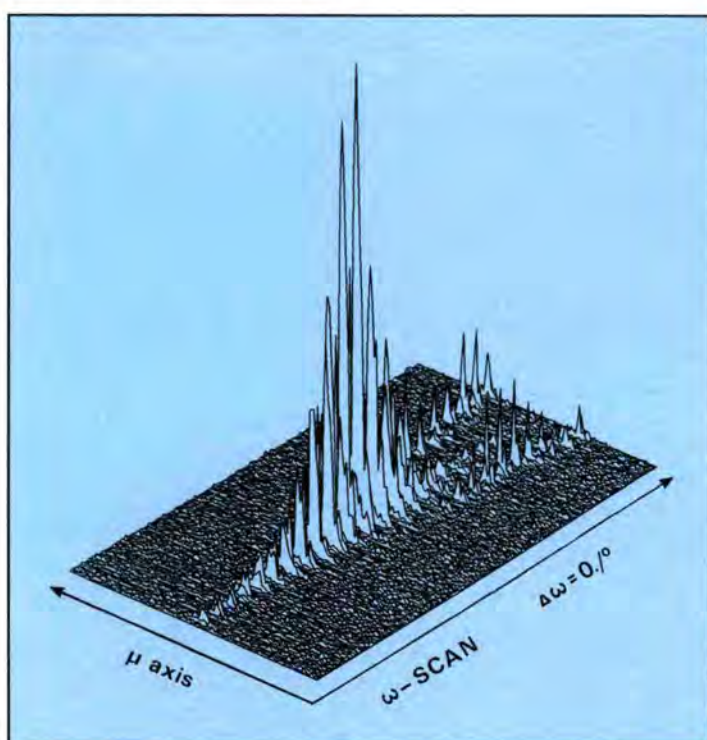


Figure 38: Recording on the D19 PSD of the scattering near 641. The crystal is rotated around a vertical axis,  $\omega$ , and the  $\mu$ -axis is the vertical direction of the detector. In all there are 25 stills at intervals of  $0.1^\circ$ . The large peak to the left is non-Bragg scattering, while the two small peaks are 640 and 641.

## Structures of oligosaccharides

Several successful experiments on cyclic oligosaccharides have been done this year using the D19 position sensitive detector: on a 6-residue  $\alpha$ -cyclodextrin and on an 8-residue  $\gamma$ -cyclodextrin.

A 1:1 cyclopentanone:  $\alpha$ -cyclodextrin inclusion complex has been studied at room temperature and at 20K. The complex is unusual in that the cyclodextrin has hexagonal symmetry. The included molecule remains disordered at low temperature, but because of the high quality of the diffraction data, it has been possible to determine the predominant conformer (collaboration with University of Paris).

A large deuterated crystal of  $\gamma$ -cyclodextrin  $\cdot 14 \text{H}_2\text{O}$  was studied at room temperature and at 110K. The hydrogen bonding scheme in this system has been compared with that in the non-deuterated 11-hydrate measured carefully at Oak Ridge (collaboration with University of Berlin).

## Clathrate hydrates

Clathrate hydrates constitute a class of solids in which the guest molecules or atoms occupy holes in icelike lattices made up of hydrogen-bonded water molecules; the usually unstable empty clathrate is stabilized by inclusion of those guest species. These compounds are interesting for several reasons. Much can be learned about the water-water interactions in these topologically rather complex systems: clathrate structures have served as models for nucleation phenomena and for cluster stability in molecular beam systems. Likewise much can be learned about the guest-host interactions (which affect e.g. dramatically the thermal conductivity) with a wide range of possible guest species ranging from noble gas atoms to large organic polar molecules. Up to now the simplest hydrates were thought to be formed by argon, krypton and xenon enclathrated in a relatively complex cubic icelike lattice. In recent experiments performed by a group from Birkbeck College and ILL the hydrate of helium has been obtained for the first time and its structure has been determined and refined (see Fig. 39). It is formed at helium gas pressures exceeding 0.28 GPa and its host lattice resembles the lattice of ordered ice II. This allows for the first time a comparison between the empty clathrate and its gas loaded counterpart. The helium content can be varied with the applied helium pressure and follows an ideal solution behaviour with a Langmuir constant of  $0.07(1) \text{ GPa}^{-1}$  and a limiting composition of  $\text{He} \cdot 6\text{H}_2\text{O}$ . It has been found that the helium enclathration stabilizes the ice II-line host lattice and prevents the formation of ice III, V and IX, thus changing radically the phase transformation behaviour in this part of the phase diagram.

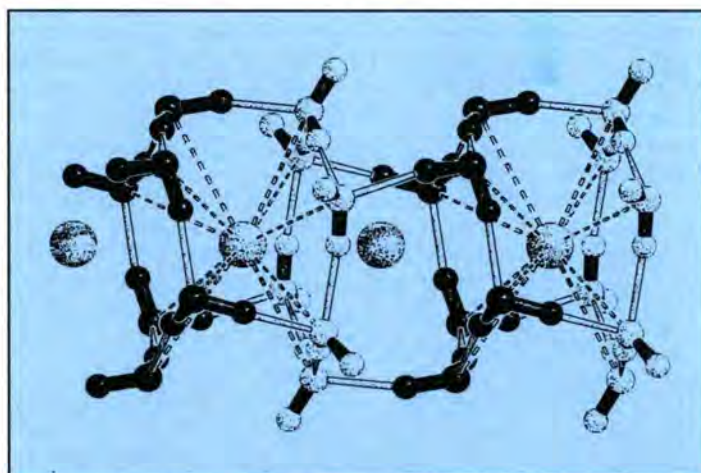


Figure 39: He clathrate in an Ice II like lattice.

## Ab-initio structure solution from powders

An interesting example of ab-initio structure solution from powder diffraction data using the new high-resolution instrument D2B has been the recent determination of the low-temperature structure of  $\text{SF}_6$ . This simple octahedral molecule has been the subject of extensive molecular dynamics simulations which demonstrated the orientational-disorder in the cubic phase. The molecular dynamics simulations predict the presence of an intermediate trigonal phase, followed by a further transition to a triclinic structure, space group P1 with  $Z=3$ . Measurement of the diffraction pattern as a function of temperature on D1B showed only a single transition at about 95K on warming the sample from 5K to 180K. Analysis of the high-resolution diffraction patterns obtained on D2B of  $\text{SF}_6$

at 5K and 60K showed that the correct spacegroup of the low-temperature phase is  $C2/m$  with  $Z=6$ . The existence of two crystallographically distinct  $SF_6$  molecules (shown in figure 40) in the ratio 1:2 in the low-temperature structure is in good agreement with spectroscopic studies.

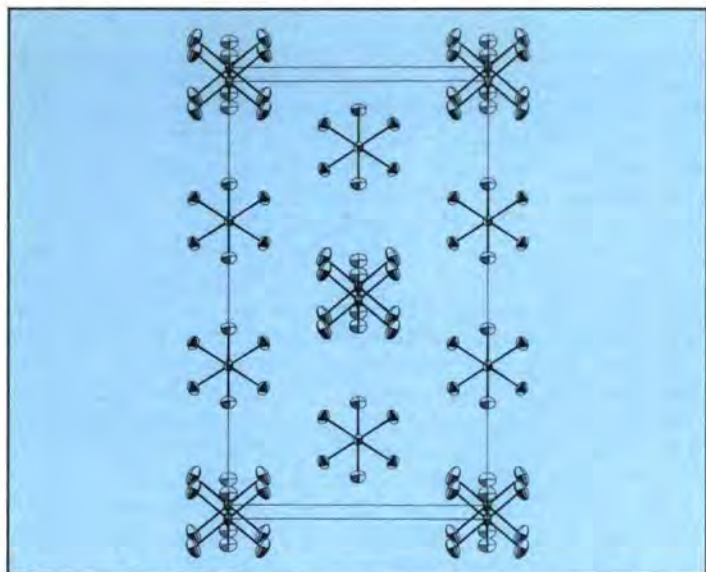


Figure 40: Plot of the monoclinic structure of  $SF_6$  at 60 K showing the two types of molecules and their molecular librations.

Further work on combining X-ray and neutron data in structure solutions has also been carried out. A good example is the structure analysis of lead oxalate,  $PbC_2O_4$ , which crystallizes in space group  $P\bar{1}$  with two independent oxalate ions (collaboration between Aarhus University, Brookhaven National Laboratory and ILL). The unit cell was determined from Guinier photographs. After this the lead atom was located using data extracted from a synchrotron powder profile recorded at the NSLS, followed by determination of the light atom positions employing difference Fourier techniques. Finally, because the scattering of lead strongly dominates the X-ray spectrum, the structure was confirmed by a Rietveld refinement using data from D2B. It was gratifying to notice, though, that even in the presence of lead, the light atoms could be determined from the X-ray data with a precision of 0.02 Å.

One commonly occurring problem in very high resolution powder work is broadening from grain size, strain or stacking fault effects, and in the present case the X-ray data showed very highly anisotropic broadening. Eventually this could be related to the crystal arrangement, which consists of alternating layers of lead and oxalate ions. In the lead layer slips along the lead-lead direction seem to occur, creating peak broadening in the corresponding Bragg reflections. In addition this could lead to irregularities in the direction orthogonal to the layers, and also here peak broadening is found. High resolution powder data might therefore simultaneously give the solution of non-trivial structural problems and some indication of the nature of the imperfections in the long range order.

### Charge density studies

For a number of years a collaboration for the study of sulphur-oxygen contacts and bonds has existed between the physics department of the University of Grenoble and the ILL, involving a different stages collaboration with the CNRS in Toulouse, University of Caen, the Hungarian Academy of Science and the Weizman Institute of Science. Most recently

the structure of 2,5-diaza-1,6-dioxo-6a-thiapentalene,  $C_3H_2O_2N_2S$ , was studied using X-ray and neutron data, recorded at 122K. Many of the usual features are found, such as density accumulation in carbon-carbon and carbon-nitrogen bonds. An almost complete absence of density outside the molecule at the oxygen position was however very puzzling, and in disagreement with electron densities obtained by local-density-functional quantum chemical calculations, which show large positive density at the oxygen "lone-pair" positions. In order to resolve this puzzle, further X-ray measurements at 10K have therefore been carried out in collaboration with Aarhus University, and the analysis is in progress. The result of this study is given in figure 41, showing the electron deformation density, defined as the difference between the observed density and a theoretical density made from spherical, non-bonding atoms. The density at the oxygen position is this time increased, improving the agreement between observed and calculated densities.

### Advanced data evaluation

For more than three years close collaboration now exists between statistics experts of the University of St Etienne, the Computing Department of the ILL and several members of the college V. This presently leads to a series of lecture courses for physicists given by A. Antoniadis on the statistical analysis methods, and to a research and development project for more performing algorithms and softwares for the neutron diffraction data treatment and analysis.

Recent practical results of this work are:

- "ABFfit": a software for the profile analysis of powder diffraction patterns by means of a maximum likelihood algorithm. This provides highly interactive graphical facilities for processing of individual patterns, but may also run in the automatic mode for the treatment of large series of smoothly changing patterns.
- a new vectorial least-squares algorithm.

The maximum likelihood algorithm developed for exponential families of distributions (e.g. ABFfit) is of the non-linear type. The effort is presently centered around the search for a measure of the non-linearity in the models which are of a practical use for the neutron pattern analysis. This will allow to establish and/or to correct for the various interferences in the fitted parameters.

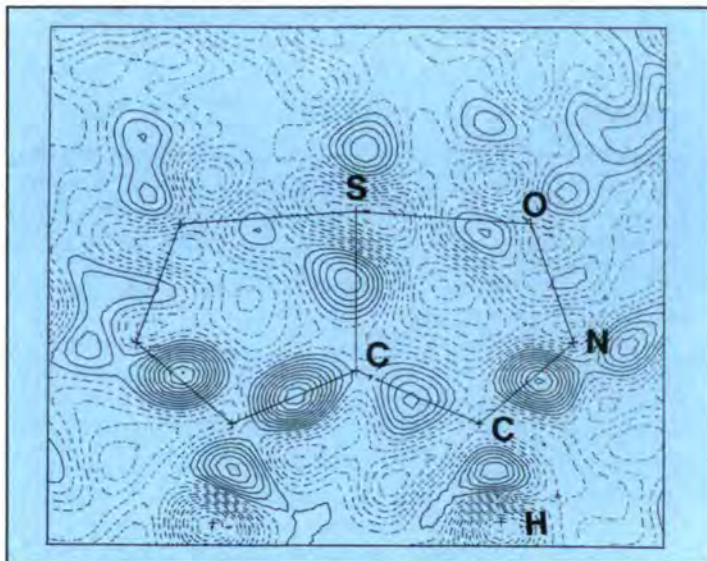


Figure 41: Deformation density for  $C_3H_2O_2N_2S$  through the plane of the molecule. Contours at 0.05  $e/\text{Å}^3$  intervals, with negative contours dotted.

## Crystallography of magnetic systems

### Low dimensional magnetic properties of USOVITE-type compounds

The natural usovite  $\text{Ba}_2\text{CaMgAl}_2\text{F}_{14}$  is characterized by a layer type structure with the general formula  $\text{Ba}_2(\text{M}^{2+})\text{M}^{2+}(\text{M}^{3+})_2\text{F}_{14}$ . The diamagnetic cations can be replaced by magnetic ones leading to the formation of chains, layers or trimeric units of magnetic ions with very original magnetic properties. For example, if  $\text{M}^{2+}$  and  $\text{M}^{2+}$  are paramagnetic species, and  $\text{M}^{3+}$  is a diamagnetic one, the magnetic ions constitute isolated chains parallel to the [101] direction (figure 42); this is the case for  $\text{Ba}_2\text{Mn}_2\text{Al}_2\text{F}_{14}$ . In this compound the model used to calculate the susceptibility is based on classical spins and takes into account the different behaviour of the two types of sites. The three dimensional magnetic ordering (3D) occurs at  $T_N = 1.65\text{K}$  as determined on D1B. The magnetic structure has been determined, the thermodiffractogram is shown on figure 43.

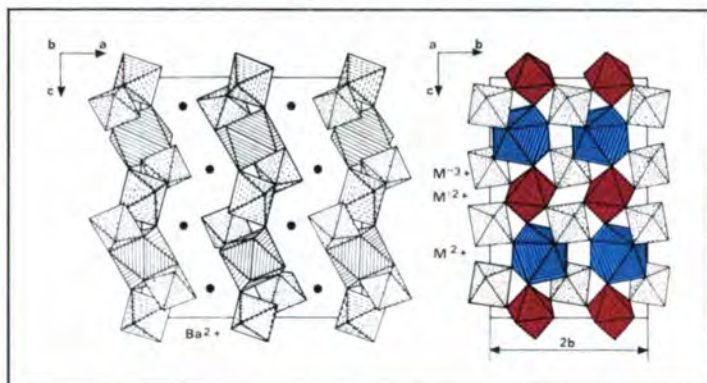


Figure 42: Projections of the USOVITE structure.

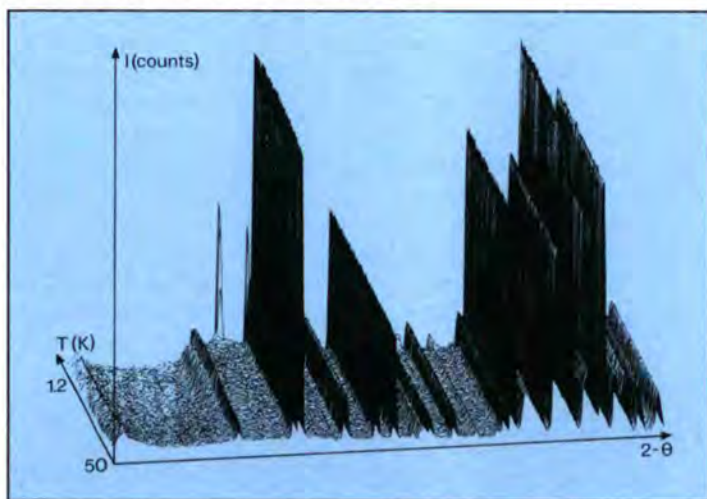


Figure 43: Thermodiffractogram of  $\text{Ba}_2\text{Mn}_2\text{Al}_2\text{F}_{14}$  observed on D1B.

When  $\text{M}^{2+}$  is a diamagnetic cation such as  $\text{Ca}^{2+}$ , the  $\text{M}^{2+}$  and  $\text{M}^{3+}$  paramagnetic ions form a lozenge chain linked by corners parallel to the [010] direction. This cationic distribution has been achieved in  $\text{Ba}_2\text{CaM}^{2+}\text{Fe}_2\text{F}_{14}$  ( $\text{M}^{2+} = \text{Mn, Co}$  and  $\text{Cu}$ ). For the Mn and Co compounds the magnetic behaviour is characteristic of ferrimagnetic chains. The thermodynamic properties of the exchange couplings in this type of chain have been calculated from a classical point of view. The magnetic structures have been determined. The 3D magnetic ordering has been observed at  $T_N = 35\text{K}$  for the Co compound, and at  $T_N = 26\text{K}$  for the Mn compound.

In the case of  $\text{Ba}_2\text{CaCuFe}_2\text{F}_{14}$ , the Jahn-Teller effect of the  $\text{Cu}^{2+}$  cation ( $d^9$ ) leads to the formation of linear trimeric magnetic units ( $\text{Fe}^{3+} - \text{Cu}^{2+} - \text{Fe}^{3+}$ ). The model to calculate the susceptibility is based on a Heisenberg hamiltonian with different g values for the  $\text{Fe}^{3+}$  and  $\text{Cu}^{2+}$  ions. The 3D magnetic ordering has been observed at  $T_N = 2.6\text{K}$ .

In all these compounds magnetic correlations of low dimensionality (2D or 1D) are present well above the ordering temperature. They appear in the diffractograms as bumps in the background.

### CRYOPAD: A new technique for generalised polarisation analysis

The theory of polarised neutron scattering, and of the polarisation of the scattered beam, was first presented by Halpern and Johnson (Phys. Rev. 55, 898 (1939)). They showed that only components of the magnetisation perpendicular to the scattering vector will give rise to magnetic scattering, and that the neutron polarisation direction is rotated on scattering by components of magnetisation not parallel to its initial direction. The theory was further developed by Blume (Phys. Rev. 130, 1670 (1963)). Since both the initial and final polarisations are vector quantities a complete polarisation analysis experiment should define all three components of both vectors. However up to now generalised polarisation analysis has only been routinely applied in transmission studies of domain structure and kinetics (Reckvelde, J. de Physique 32 C579 (1971)); in most scattering experiments only a single component of the scattered polarisation has been analysed as in the experimental arrangement of Moon, Riste and Koehler (Phys. Rev. 181 920 (1969)).

A new experimental technique: CRYOPAD (Cryogenic Polarisation Analysis Device) which enables all three components of both the incident and scattered polarisation to be determined has been developed at the ILL. It has been used in the first instance to study fine details on the magnetic structure of  $\text{Cr}_2\text{O}_3$ . The essential feature of a device to define all three components of the incident and scattered polarisation is complete control of the magnetic fields through which the neutron beam passes. In the present experimental arrangement we have exploited the Meissner effect to isolate regions in which different and well defined magnetic fields are present. The CRYOPAD makes use of two superconducting screens, the Inner Meissner Screen (IMS) and the Outer Meissner Screen (OMS). Both screens have the form of long vertical test-tubes; they differ in diameter by a factor of 2 [fig. 44]. The IMS is inserted coaxially into the OMS, so that the scattering plane is divided radially into three regions: the Central Region (CR), the annular Intermediate Region (IR) and the Outer Region (OR). The cylindrical geometry and the extremely effective magnetic screening (up to several 100 Oe) of the Meissner effect makes these three regions magnetically independent. Thus each region can be dedicated to control a different spherical coordinate of the neutron polarisation.

The CRYOPAD was installed at the sample position of the polarised triple axis spectrometer IN20 at ILL. To test its effectiveness some measurements were made on nuclear and magnetic reflections from  $\text{MnF}_2$  which confirmed the expected action of the adiabatic spin-field rotators (ASFR) placed outside the cryostat. For the experiment on  $\text{Cr}_2\text{O}_3$  a crystal was mounted with the [010] (hexagonal) axis vertical. Measurements were made on the 102 and 104 reflections at temperatures of 7, 110, 150, 255, 310 and 320K. At each temperature and for each reflection the scattered polarisation direction was found for each of three incident directions.

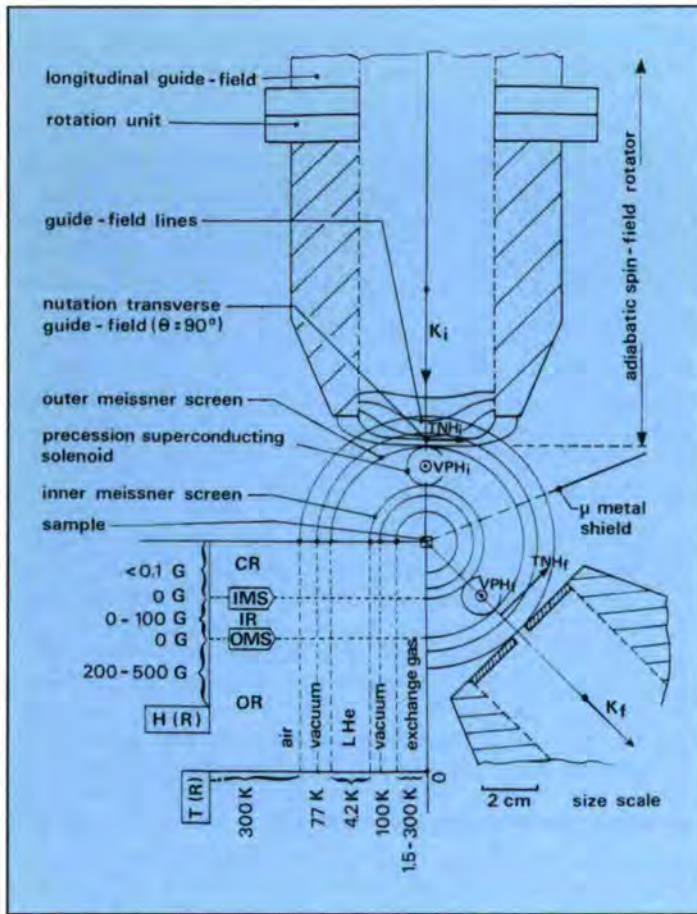


Figure 44: Schematic horizontal section of the CRYOPAD showing the magnetic fields in the Meissner screens and independent regions; CR: central region, IMS: internal Meissner screen, IR: intermediate region, OMS: outer Meissner screen, OR: outer region. Other notations are:  $K$  neutron wave-vector,  $TNH$  transverse nutation guide field,  $VPH$  vertical precession coil; subscripted  $i$  for the incident and  $f$  for the scattered beams. The lower left hand corner gives part of a vertical section showing the temperatures of the various screens.

The magnetic structure factors for the collinear magnetic structure, described on hexagonal axes, have the form

$$\underline{M}(\underline{\kappa}) = 12i\mu_{Cr} \hat{S} f(\kappa)\sin(2\pi lz) \text{ for } l \text{ even,} \quad (1)$$

and are zero for  $l$  odd, where  $\mu_{Cr}$  is the magnetic moment,  $\hat{s}$  a unit vector parallel to the spin on the atom at  $(00z)$ ,  $f(\kappa)$  the form factor and  $z = 0.3475$ . The magnetic interaction vectors  $\underline{Q}(\underline{\kappa}) = \underline{\kappa} \times \underline{M}(\underline{\kappa}) \times \underline{\kappa}$  are therefore pure imaginary ( $\underline{Q} = -\underline{Q}^*$ ) and lie in the plane perpendicular to  $\underline{\kappa}$  containing the spin direction  $\hat{s}$ .

For elastic scattering by a Bragg reflection the expression given by Blume for the scattered polarisation can be written:

$$\underline{P}_s \delta\sigma/\delta\omega = \underline{P}_i (N N^* - \underline{Q} \cdot \underline{Q}^*) + \underline{Q} N^* + \underline{Q}^* N + \underline{Q} \cdot \underline{P}_i \cdot \underline{Q}^* + \underline{Q}^* (\underline{P}_i \cdot \underline{Q}) - i \underline{P}_i \times (\underline{Q} N^* - \underline{Q}^* N) + i (\underline{Q} \times \underline{Q}^*) \quad (2)$$

where  $N$  is the nuclear structure factor,  $\underline{Q}$  the magnetic interaction vector described above, and  $\underline{P}_i$  and  $\underline{P}_s$  are the incident and scattered polarisations respectively. For  $Cr_2O_3$  for which  $N$  is real and  $\underline{Q}$  imaginary (2) reduces to:

$$\underline{P}_s(1 + \gamma^2) = \underline{P}_i(1 - \gamma^2) + 2 \hat{q} \gamma^2 \underline{P}_i \cdot \hat{q} + 2 \underline{P}_i \times \hat{q} \gamma \quad (3)$$

where  $\hat{q}$  is a unit vector parallel to  $\underline{Q}$  and  $\gamma = |\underline{Q}|/N$

Defining orthogonal axes  $x$  parallel to  $\underline{\kappa}$ ,  $z$  to  $[010]$  and  $y$  parallel to  $\hat{q}$  from (3) the  $x$ ,  $y$  and  $z$  components of the scattered polarisation are then given by

$$P_{sx} = (P_{ix}(1 - \gamma^2) - 2P_{iz}\gamma)/(1 + \gamma^2)$$

$$P_{sy} = P_{iy}$$

$$P_{sz} = (P_{iz}(1 - \gamma^2) + 2P_{ix}\gamma)/(1 + \gamma^2) \quad (5)$$

These equations show that whilst the component of polarisation parallel to  $\hat{q}$  is not changed by scattering the other component is rotated in the plane perpendicular to  $\hat{q}$  in a direction which depends on the sign of  $\gamma$  and by an amount which depends on its magnitude.

The sign of  $\gamma$  depends on the choice of the spin direction (either parallel or antiparallel to  $[001]$  on the atom at  $(00z)$  i.e. the direction of the spin with respect to its non-centrosymmetric environment. Because of this asymmetry the two different orientations are distinguishable and should have different energies. Determination of the sense of the rotation of the polarisation therefore allows the lower energy configuration to be determined. If the two configurations have nearly equal energies, different parts of the crystal may correspond to different configurations, in which case rotations in different parts of the crystal take place in different directions giving rise to some depolarisation of the scattered beam.

The measurements at 7K of the 102 and  $\bar{1}04$  reflections gave values of  $\gamma_{102} = 2.13(1)$ ,  $\gamma_{\bar{1}04} = 1.032(5)$  and the fraction  $\alpha$  of the crystal in the state with the  $Cr^{3+}$  ion at  $(0,0,0.347)$  pointing along  $[001]$  as 0.86 (2). However the results show that the depolarisation by the 102 reflection is greater than that by the  $\bar{1}04$ , whereas the simple theory predicts that the maximum depolarisation should occur when  $\gamma = 1$ . Measurements of the 102 reflection as a function of temperature show that indeed the polarisation decreases as the ordered moment decreases and  $\gamma$  approaches unity; while by contrast the polarisation of the  $\bar{1}04$  reflection is a continually increasing function of temperature, consistent with  $\gamma$  decreasing continuously from its value of approximately unity at 7K. These observations require that the source of depolarisation is a property of the magnetic structure factor itself and is different for the 102 and  $\bar{1}04$  reflections rather than being due to domain effects which are a property of the crystal as a whole.

In  $Cr_2O_3$  the  $Cr^{3+}$  ion is coordinated by an octahedron of oxygen ions which has trigonal symmetry and is made up of two equilateral triangles between which the  $Cr^{3+}$  ion is sandwiched, one of these triangles is 13% smaller than the other and is 30% further away from the central ion. Our results allow us to conclude that the energetically favourable moment orientation in  $Cr_2O_3$  is with the  $Cr^{3+}$  spins pointing towards this smaller triangle.

### Polarised neutrons; Powder diffraction

The option of polarised neutrons is now available on D1B. Using a supermirror to polarize the neutrons one obtains an incoming beam of 97% polarised neutrons at 10% of the initial flux ( $\lambda = 2.52 \text{ \AA}$ ). A  $\pi$ -coil can be used to flip the polarised neutrons. The sample can be cooled down to 4K within a magnetic field of up to 1 Tesla.

The intensity of the spin up neutrons is

$$I_{\uparrow} \sim N^2 + M^2 + 2NMP_i$$

where  $N$  and  $M$  correspond to the nuclear and the magnetic moment and  $P_i$  is the polarisation.

For flipped neutrons:

$$I_{\downarrow} \sim N^2 + M^2 - 2\mu MP_i (2E - 1)$$

with  $E$  being the flipper efficiency.

The flipping ratio is

$$R = \frac{I\uparrow}{I\downarrow} = \frac{1 + 2P_i\gamma + \gamma^2}{1 - 2P_i\gamma + \gamma^2}$$

with  $\gamma = \frac{N}{M}$  and  $e = 2E-1$

R being known from the experiment (Integrated Intensities of  $I\uparrow$  and  $I\downarrow$  for every peak),  $P_i$  and  $e$  from the initial optimisation of the set-up using a Heussler single crystal,  $\gamma$  can then be calculated.

The difference plot  $I\uparrow - I\downarrow = 2NMP_i(1 + e)$  has positive and negative peaks. Knowing approximately the nuclear structure factors of the reflections these peak intensities can be directly related to the orientation of the magnetic spins at the different sites. Without bothering about absolute values of the magnetic spins this presents an easy and fast method to discriminate for instance between ferro- and ferrimagnetic models.

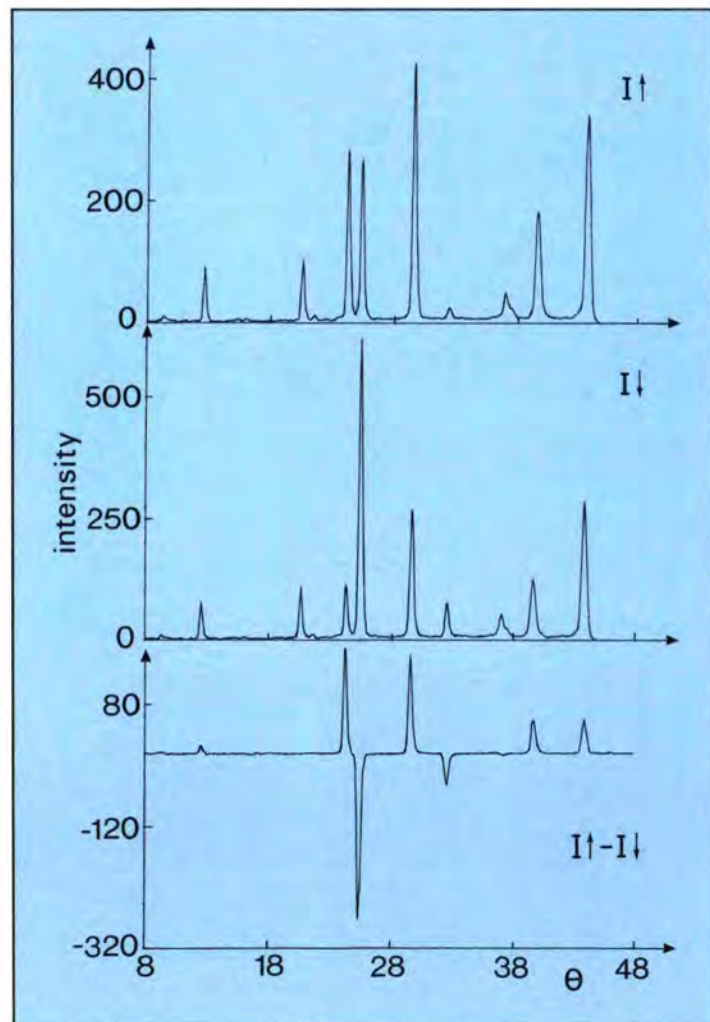


Figure 45: Spin-up  $I\uparrow$ , spin down  $I\downarrow$  and difference for  $CuCr_2Se_4$ , recorded on D1B.

Absolute values of  $N$  and  $M$  can be determined by measuring as well with “depolarised” neutrons (one uses a piece of soft iron to depolarise the polarised beam in order not to change the set-up of the experiment) which allows to extract the scale-factor from a purely nuclear or purely magnetic peak.

$$I_{DEPO} = K \cdot j \cdot LPJ \cdot (N^2 + M^2) \text{ with } N \gg M \text{ or } M \gg N$$

$\downarrow \quad \downarrow \quad \downarrow$   
 Scale factor    Mul- tiplicity    Lorentz factor

As an example for the usefulness of this new option fig. 45 shows the pattern and difference of  $I\uparrow$  and  $I\downarrow$  as measured for the spinel sulfide  $CuCr_2Se_4$ . Just from the qualitative evaluation of the data it was concluded that the Copper ions have no magnetic moment while there is a localised magnetic spin on some of the chalcogen atoms with an orientation opposite (ferrimagnetic) to that of the chromium spin moment.

### Observation of the helimagnetic-ferromagnetic phase coexistence in terbium by neutron diffraction topography

The way two magnetic ordered phases spatially coexist in a given single crystal sample lead to a rich variety of situations which were scarcely investigated from an experimental point of view. The observation of the shape and evolution of the interface, as well as the unambiguous identification of the magnetic phases, can be achieved by using neutron diffraction topography.

An example of this approach, the helimagnetic-ferromagnetic phase coexistence in a rectangular platelet shaped crystal of terbium as a function of the applied field, is shown in fig. 46. The 0002- $\tau$  magnetic satellite was used to perform the topographs, implying that only the helimagnetic regions contribute to the diffraction (white areas on the photographs). Most of the interfaces are parallel to both the applied field and the magnetization in the ferromagnetic regions in order to minimize the term of the energy which is associated with the demagnetizing field. On the other hand ferromagnetic areas occupy in a predominant way the two sides of the sample, the center being finely divided between both phases. This arrangement, added to a more complex pattern in the close neighbourhood of the two edges perpendicular to the field, results in a nearly constant internal field, equal to the critical field.

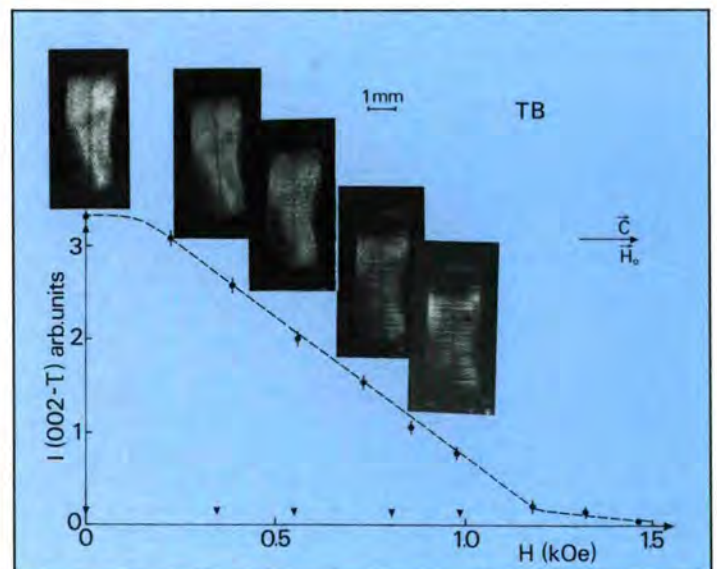


Figure 46: Field driven helimagnetic-ferromagnetic transition in a platelet-shaped single crystal of Tb. The topographs show the evolution of the helimagnetic area.

The same helimagnetic-ferromagnetic transition occurs very differently when considering the temperature driven case (fig. 47): the magnetostatic energy term is, indeed, very reduced by the presence of domains in the ferromagnetic phase, and other terms of energy (elastic, interface,...) dominate the coexistence, which is reproduced over several thermal cycles.

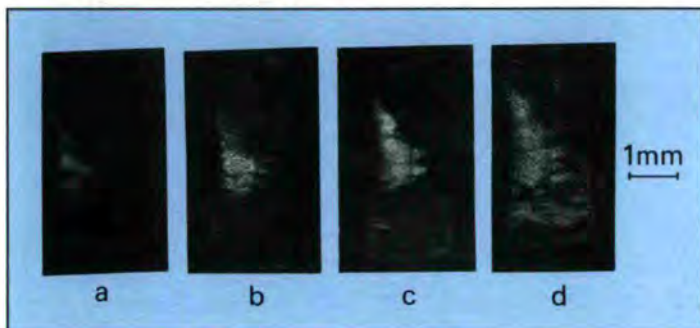


Figure 47: Four stages of the temperature driven helimagnetic-ferromagnetic transition in Tb ( $T = 221K$ ). The helimagnetic region is white. Its intensity corresponds to  
 a) 0.07  
 b) 0.32  
 c) 0.71  
 d) 0.88 of the maximum intensity of the 0002- $\tau$  reflection used to perform the topographs.

### The multiphase transition of MnRhAs

In the series of the  $MM'X$  compounds (M, M' 3d or 4d metals and X: As or P), MnRhAs (hexagonal, space group  $P6_32m$ ) is one of those that exhibit the most complex magnetic behaviour. Above the 3D ordering temperature ( $T_N = 238K$ ) and up to  $3T_N$ , strong magnetic correlations have been observed. Below this temperature the ordered state may be described by stackings of ferromagnetic (00l) planes corresponding to a propagation vector  $q_1 = [001/2]$ . Below 190K a canting of the magnetic moments occurs giving rise to a ferromagnetic component parallel to c. In a temperature range of 15K centered on 150K a magnetic reordering takes place towards another antiferromagnetic phase. This low temperature phase is based on the stacking of the same ferromagnetic (00l) planes as those of the high temperature phase but the sequence corresponds to a propagation vector  $q_2 = [001/4]$ .

Neutron diffraction performed on powder samples not only shows in the reordering temperature range ( $q_1 = [001/2] \rightleftharpoons q_2 = [001/4]$ ), the coexistence of the peaks associated to  $q_1$  and  $q_2$  but also indicates that additional magnetic phases are present. An investigation of this puzzling behaviour was then undertaken on a carefully selected platelet shaped single crystal, at  $\lambda = 2.4\text{\AA}$  (a pyrolytic graphite filter was used to suppress  $\lambda/2$  contamination) on the S20 instrument, in order to obtain a good separation of all the peaks and perform topographs of the different phases.

Figure 48 shows  $(\omega-2\theta)$  scans of the reciprocal c direction recorded at several temperatures within the coexistence temperature range: the magnetic reordering (from  $q_1$  to  $q_2$ ) is associated to the occurrence of at least two sets of new magnetic peaks which could be indexed on one hand with a  $q_3 = [001/6]$  propagation vector and on the other hand as  $(00((2n+1)/4) \pm \tau)$  with  $\tau$  varying with the temperature and such that  $0 < \tau < 1/12$ . The width of these last peaks (more than twice that of the other peaks) and their rather surprising indexation (satellites of the magnetic superstructure peaks) lead to the conclusion that they probably correspond to an extended boundary between the phases. Neutron topographs were

recorded in the phase coexistence temperature range and show clearly that the whole crystal surface cannot be completely restored with only the scattering areas corresponding to  $q_1$  and  $q_2$  (figure 49). The missing areas are those associated with the longer periods. Preliminary topographs of these peaks, although not establishing an unambiguous spatial connection with the two other phases, clearly indicate that the  $q_3$  zones are distributed around the  $q_1$  ones or occupy the regions just left by this phase.

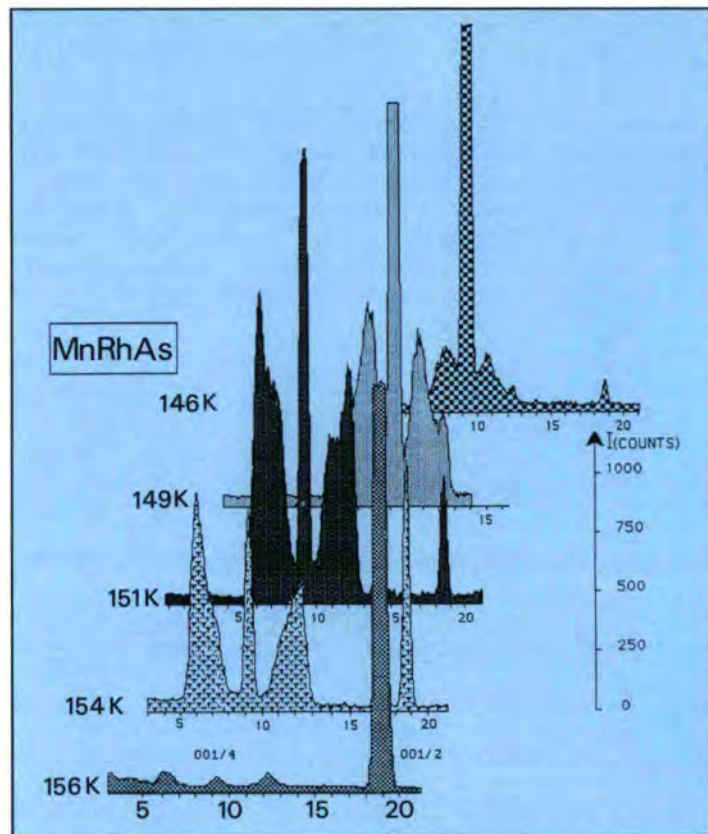


Figure 48: Evolution of the  $(\omega-2\theta)$  scans of the [001] reciprocal direction recorded in the phase coexistence temperature range in MnRhAs. The scans have been performed in the vicinity of the (001/4) reflection. The reflections apart (001/4) are due to longer period magnetic phases (see text).

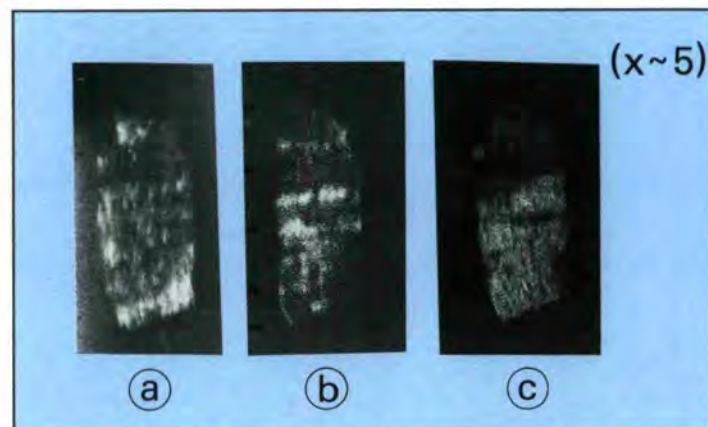


Figure 49: Neutron topographs recorded at 156.2K in the phase coexistence temperature range of MnRhAs: (a) (003/4) and (c) (001). The topograph performed using a given magnetic peak only displays the image of the regions occupied by the corresponding magnetic phase. Topographs (a) and (b) show that the whole crystal surface cannot be completely restored with the scattering areas related to  $q_1 = [001/2]$  and  $q_2 = [001/4]$ .

Secretaries: M. Lehmann, J.Pannetier

## Schwinger Scattering

As is well known, the principal terms in the cross-section for scattering of neutron beams from matter are the strong interaction between the neutron and the atomic nuclei, and the magnetic dipole interaction between the neutron magnetic moment and the magnetic field due to any unpaired electrons. There are however in addition to these strong interactions several much weaker interactions giving rise to small additional contributions to the scattering cross-section. One of the most interesting of these is the Schwinger or "spin-orbit" interaction (1). Schwinger scattering arises from the relative motion of the neutron and the electric field of the electronic and nuclear charges on the atoms. The sense of the interaction is changed depending on whether the neutron is moving towards or away from an atom, and hence is antisymmetric with respect to the atomic nuclei. This gives rise to scattering in quadrature with the nuclear scattering from the same nucleus ; represented by an imaginary contribution to the scattering amplitude.

The contribution  $X_s$  of Schwinger scattering to the total structure factor is given by (2):

$$X_s = i \frac{\gamma e^2}{mc^2} \frac{m}{m_0} \frac{(\underline{k} \times \underline{k}') \cdot \underline{P}}{|\kappa|^2} F_s = 0.295i \frac{\cot\theta}{2} F_s (\hat{\underline{v}} \cdot \underline{P}) \times 10^{-3}$$

where  $\gamma$  is the neutron magnetic moment in nuclear magnetons,  $m$  and  $m_0$  the electron and neutron masses respectively,  $\underline{k}$  and  $\underline{k}'$  the incident and scattered wave-vectors and  $\kappa$  the scattering vector ;  $\underline{P}$  is the polarisation vector and  $\hat{\underline{v}}$  a unit vector perpendicular to the plane of  $\underline{k}$  and  $\underline{k}'$ .  $F_s$  is the Schwinger structure factor:

$$F_s = \sum_j f_{sj}(\kappa) \exp(\underline{\kappa} \cdot \underline{r}_j) \text{ with } f_{sj}(\kappa) = Z_j - f_j(\kappa)$$

the summation being over all the atoms (subscripted  $j$ ) in the unit cell. The rest of the symbols have their usual meanings. The polarisation dependence of the scattering arises because the interaction is between the neutron magnetic moment and the apparent orbital motion of the atomic charges as seen by the neutron. It is this polarisation dependence which makes it possible to observe the Schwinger scattering in particular cases even though its contribution to the cross-section is less by a factor of the square of the ratio of the electron to neutron masses with respect to the strong scattering processes.

Observation of the Schwinger scattering with unpolarised neutrons is only possible in very special cases, such as that of isotopically enriched tungsten crystals (3) for which the nuclear scattering is nearly zero. With polarised neutrons however the Schwinger scattering may become measurable by interference with any imaginary component in the strong scattering processes. Such imaginary terms arise in centrosymmetric structures from the imaginary parts of the nuclear scattering lengths. Measurement of the polarisation dependence of the cross-section permits determination of these imaginary scattering lengths which has enabled tests of the validity of the Breit-Wigner formula to be made (4,5). In non-centrosymmetric crystals such as GaAs the Schwinger effect gives rise to quite significant polarisation ratios, and though the dependence of  $F_s$  (defined above) on the charge distribution in the atoms enables information on bonding charge distributions to be obtained (6).

J. Brown

[1] J. Schwinger, *Phys. Rev.* 73 407 (1948)

[2] M. Blume, *Phys. Rev.* 133 A1367 (1964).

[3] Yu. A. Alexandrov, L.N. Sedlakova, B. Chalupa, R.T. Michalec, P. Micula and M. Vrana, *Phys. Letts.* 72A 361 (1979).

[6] P.J. Brown and J.B. Forsyth, *Conference on Neutron Scattering Sidney Australia, August 1987.*

# COLLEGE 6 LIQUIDS, DISORDERED AND DEFECT MATERIALS

## Members of the College

A. Barnes	J. Lopez-Diosdado
J.P. Beaufils	A. Magerl
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J. Cook	M. Maret
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## Summary

The research activities of College VI in 1987 have covered a wide field of subjects similar to previous years ranging from quantum liquids, simple liquids, liquid alloys and low molecular liquids to amorphous materials, metal physics and diffusion of light particles in solids. The distribution of these activities on the diverse subjects, as measured by the number of proposed experiments in 1987, is shown in Fig. 50. Clearly glasses and amorphous materials have been the main subject of interest in College 6, followed by the large amount of proposals on ionic solutions, molten salts and hydrogen in metals. Regarding the load on the instruments, the largest demand in College 6 was for diffraction experiments on D4 (large range of momentum transfer up to  $24 \text{ \AA}^{-1}$ ) and for the time-of-flight (TOF) machines IN5 and IN6. Furthermore it is interesting to see that the collaboration of the college with groups of the member countries amounts about 20% of the proposals.

Although these statistical aspects are of course no appropriate yard stick with regard to the scientific value of the work carried out, it must be mentioned that the results from many experiments have again contributed to the further understanding of unresolved problems being currently of interest in the field of liquids and amorphous materials. This can be judged in detail from experimental reports and publications. In the following, only a few topics are mentioned.

The quite successful experiments on the mixed boson-fermion system  $^3\text{He}$ - $^4\text{He}$  were continued. Furthermore the method of isotope substitution has been used to determine partial structure factors and thus inter- and intra-atomic distances for a large number of liquids, liquid alloys and solutions. Diffraction

studies and inelastic measurements on quasicrystals were able to give more insight into the statics and dynamics of these materials. The activities belonging to the study of the glass transition on different types of materials, boosted by mode-coupling theories describing the glass transition as a dynamical phenomenon, were again numerous and established the appearance of additional intensity at low energies. This is due to either vibrational or relaxational modes arising near the macroscopic glass transition temperature on the timescale of neutron scattering. Hydrogen diffusion in metals at low temperatures were shown to be strongly influenced by the conduction electrons. Furthermore quasielastic neutron scattering (QNS) confirmed its unique nature in determining local and dynamic features of the conduction mechanism in ionic conductors. The studies on self-diffusion and solute atom diffusion in IVb metals, involving complicated experimental and sample preparations were successfully continued.

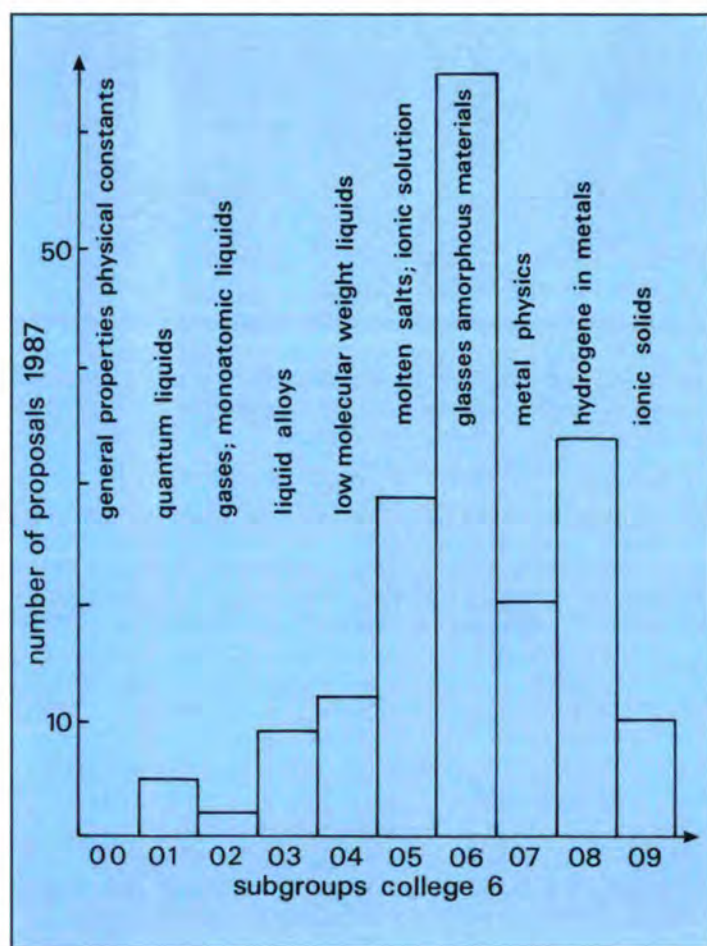


Figure 50: Research activities of College VI in 1987 (number of proposals in 1987)

## Scientific Trends and Highlights in 1987

### Quantum Liquids

The successful experiments of Scherm et al. [1] on liquid helium-3 have been followed by an investigation of the pressure-dependence of the excitation spectrum of  $^3\text{He}$ - $^4\text{He}$  mixtures [2]. The inelastic scattering from a low-concentration

mixture of  $^3\text{He}$ - $^4\text{He}$  was measured on the IN6 t.o.f. spectrometer at a range of temperatures and pressures. These results are at present being analysed and will certainly improve the knowledge of the excitations in this mixed boson-fermion system.

Two new measurements on pure  $^4\text{He}$  have been made. The IN12 triple axis spectrometer was used to determine the temperature-dependence of the full (single-plus multi-phonon) spectrum, below and above the superfluid transition point, at an applied pressure of 20 bar [3]. The aim of this experiment was to check the Woods-Svensson hypothesis [4] which effectively decomposes the spectrum into normal and superfluid components. Detailed analysis has shown significant discrepancies with the behaviour expected on the basis of this model. The Figure 51 shows maxon ( $Q = 1.13 \text{ \AA}^{-1}$ ) spectra at a range of temperatures and the corresponding one-phonon intensities.

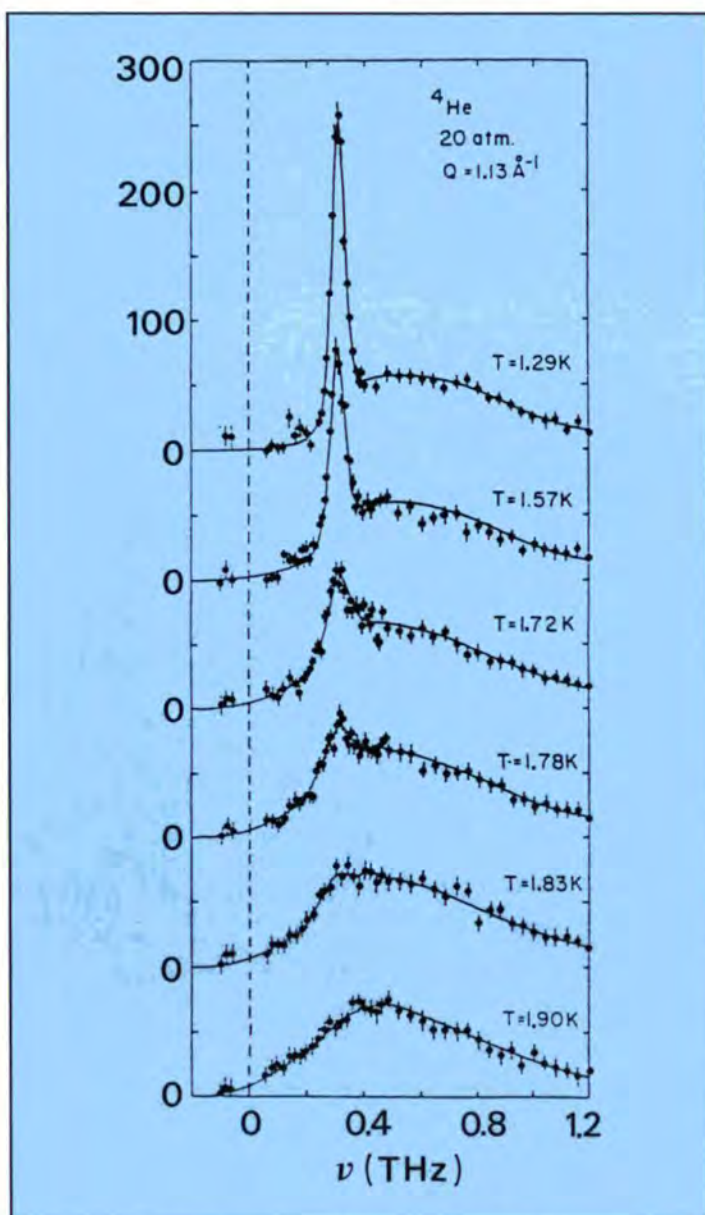


Figure 51: (a) Inelastic spectra of liquid  $^4\text{He}$  at  $P = 20$  bars, at the 'maxon' wave-vector ( $1.13 \text{ \AA}^{-1}$ ); the sharp one-phonon peak apparently disappears just at, or near  $T_\lambda$  ( $1.93\text{K}$ ).

In a second experiment, the oscillations in width and frequency [5] of the  $^4\text{He}$  scattering function at intermediate wavevectors ( $3 < Q < 12 \text{ \AA}^{-1}$ ) have been re-examined using the hot-source instrument IN1 [6]. The new data confirm the existence of the oscillations and a start has been made in determining the (non-Gaussian) shape of the scattering function.

## Structure and Dynamics of Liquids

Several studies were started on monoatomic systems, in order to obtain information on the interatomic potential from accurate measurements of the structure factor. The structure of liquid potassium determined at several temperatures up to  $700^\circ\text{C}$  led to an interesting renormalization of the alkali metal structure factors using the hard sphere diameter at the melting point [7]. Liquid gallium was measured at two temperatures on D4B in an attempt to extract the interatomic potential through the help of new computational method [8]. Gaseous  $^{36}\text{Ar}$  was measured at 140 K and four low densities ( $n = 0.9, 1.4, 1.9$  and  $2.4 \text{ nm}^{-3}$ ) for  $1.9 < k < 100 \text{ nm}^{-1}$ . The duration of each experiment varied between 3 h and 3 days as to obtain the same statistical accuracy in each of the  $H(k)$ , [ $H(k) = S(k) - 1/n$ ] (Figure 52). It is hoped that this study will show new light on the pair potential description and the extent of more-body potential effects on the structure of simple disordered systems [9].

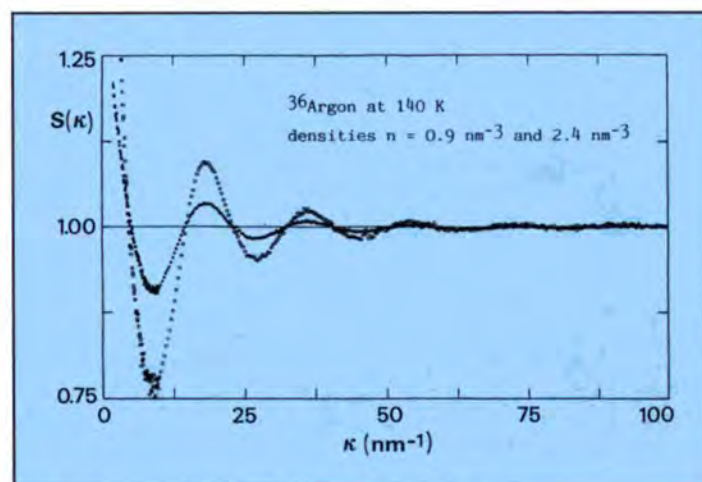


Figure 52:  $S(\kappa)$  for  $^{36}\text{Ar}$  at 140K (measurement on D4) /9/.

The analysis of TOF-experiments on liquid sodium carried out on IN5 and IN6 was able to give evidence for the validity of mode-coupling predictions. Due to the single particle motion in a liquid, shear modes are generated which counteract on the particle and lead to a slower decay of the velocity-autocorrelation function  $\langle v_i(0) v_i(t) \rangle$ . Mode coupling effects are thus expected to lead to a quasielastic line-width and peak intensity, respectively, which vary linearly with  $Q$  and a Fourier-transform of the VAF (i.e. a density of states  $z(\omega)$ ) showing a square-root cusp at  $\omega = 0$ . These predictions describe quite well the experimental results, as is shown for example in Fig. 53 for liquid sodium [10].

“Dispersion curves” measured on liquids usually show an increase with sound velocity, followed by a region where the collective excitations are overdamped due to diffusion processes. Recently a method was proposed [11] to calculate the memory function directly from the experimental  $S(Q, \omega)$  data, which

predicts that the collective excitations in alkali metals are not overdamped in the region of the maximum of the static structure factor. Measurements on liquid Cs [12] (thermal triple axes spectrometer IN8;  $k_f = \text{const} = 2.66 \text{ \AA}^{-1}$ ;  $-2\text{meV} < E < 10\text{meV}$ ; elastic resolution  $\sim 0.74\text{meV}$ ) gave an interesting preliminary result which is summarized in Fig. 54 as a dispersion curve. The increase of the dispersion curve at low Q corresponds roughly to the velocity of sound. For Q-values larger than about half the Q-value of the first maximum in  $S(Q)$  the dispersion curve decreases significantly in a range where the collective excitations are still visible as an inelastic hump in  $S(Q, \omega)$ . The maximum in the static structure factor was located at  $Q \sim 1.4 \text{ \AA}^{-1}$ .

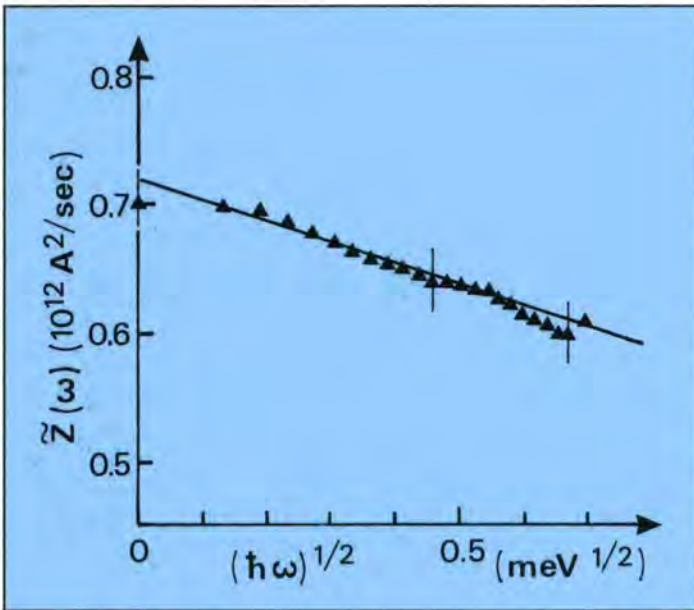


Figure 53: Density of states plotted vs  $(\hbar\omega)^{1/2}$  ( $\Delta = \text{IN6}$  measurement; straight line = least squares fit) in liquid Na [10].

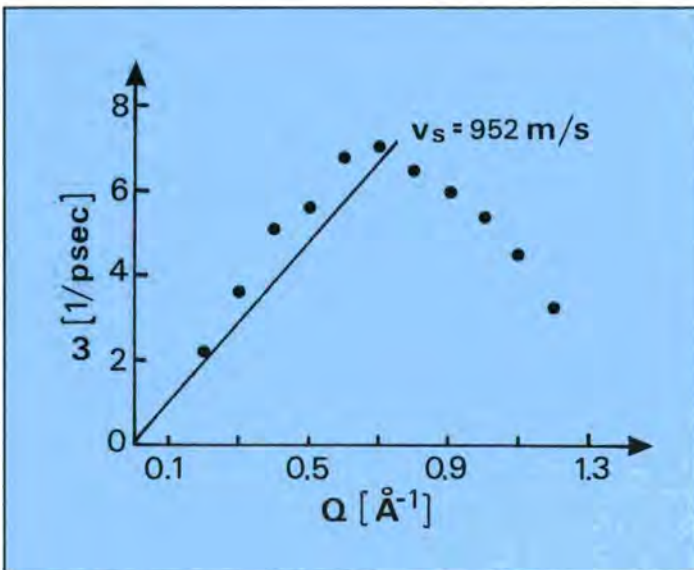


Figure 54: Dispersion relation  $\omega(Q)$  of liquid Cs at 308K (solid line: acoustic sound velocity) [12].

### Statics of Quasicrystals

The quasicrystalline, rapidly quenched alloy  $\text{Al}_{82}\text{Si}_1[\text{Mn}_x(\text{FeCr})_{1-x}]_{17}$  was studied by neutron diffraction on D4. The structure factor was directly Fourier transformed to obtain pair distribution functions. The random substitution of the FeCr  $\sigma$ -phase for Mn was unambiguously confirmed, thus allowing the extraction of Partial Pair Distribution Functions (PPDF) using the contrast variation technique (Fig. 55).

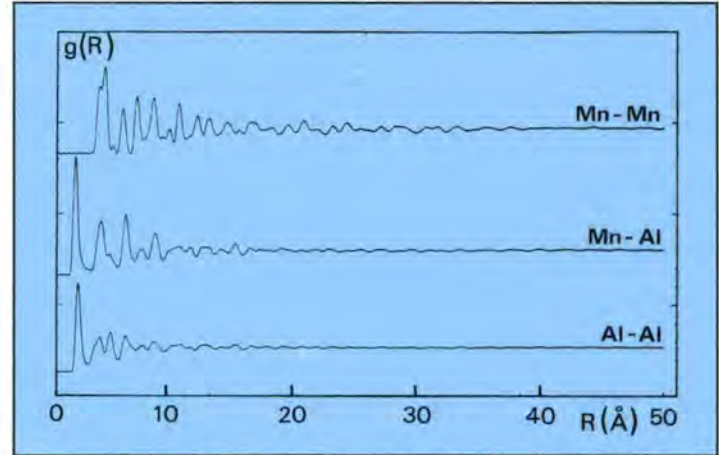


Figure 55: Partial pair distribution functions of quasicrystalline  $\text{AlSi}[\text{Mn}_x(\text{FeCr})_{1-x}]_{17}$  (Janot et al; unpubl.)

The observation of narrow and generally unsplit peaks up to quite large R values in the measured PPDF suggests that each atomic site might be a centre of inversion for the whole corresponding subnetwork. Among other interests, such a result is a post-justification of the validity of the isotropic regrouping operations included in the data reduction procedure. Close contact between transition metal atoms (T) are not observed and the smaller coordination T-T shell is split into two T-T distances at  $4.47 \text{ \AA}$  (3.3 atoms) and  $4.97 \text{ \AA}$  (6.0 atoms). Obviously enough, the T subnetwork seems to behave as the true framework of the whole structure as previously suggested.

Comparatively, the Al-Al and T-Al correlations are less ordered. With the exception of their first peak, the corresponding PPDF or PRDF exhibits rather broad features which vanish obviously at much shorter distances than their T-T partner. It appears clearly that the first coordination shell around a T atom contains only Al atoms (9.45 atoms at  $2.55 \text{ \AA}$  plus 2.35 atoms at  $3.05 \text{ \AA}$ ), while Al atoms have 10 other Al nearest-neighbours (at  $2.82 \text{ \AA}$ ) and 2.6 atoms of Mn (2 atoms at  $2.55 \text{ \AA}$  and 0.6 atoms at  $3.05 \text{ \AA}$ ). Along with high resolution powder diffraction data, the produced PPDF suggest that manganese atoms are located at vertices of a 3D Penrose network with about 15% vacant sites while the aluminum atoms would be shared by amorphous and crystalline clusters.

### Dynamics of Quasicrystals

The dynamical properties of icosahedral  $\text{Pd}_{58.8}\text{Si}_{20.6}\text{U}_{20.6}$  have been investigated in comparison with those of the metallic glass of the same composition (from which the icosahedral sample was obtained by a short annealing of the metallic glass) and of the fully crystallized sample [13]. In spite of the fact that the diffraction spectrum of icosahedral PdSiU resembles much more that of the crystallized sample (see insert in Fig. 56) the

generalized vibrational density of states (GVDOS) nearly coincides with that of the metallic glass and is very different from that of the crystallized sample. Low energy modes were detected in the GVDOS of icosahedral PdSiU like in the spectra of metallic glasses. Whether these are due to an icosahedral glass sta

te or due to phason strains is still under investigation. Also the wavelength dependence of the atomic dynamics of icosahedral PdSiU as obtained from the dynamic structure factor  $S(Q, \omega)$  resembles much more that of the metallic glass than that of the crystallized sample, showing the influence of strong atomic disorder in the icosahedral sample.

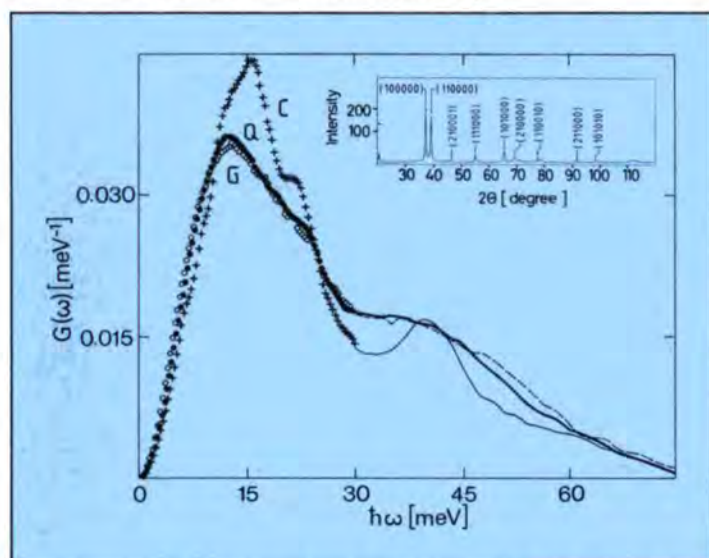


Figure 56: Generalized vibrational density of states of icosahedral (Q), glassy (G) and crystallized (C)  $\text{Pd}_{58.8}\text{Si}_{20.6}\text{U}_{20.6}$  as obtained from the sum of all TOF spectra measured on IN6 at 296K with an incident energy of 4.8meV [13].

## Dynamics of the Glass Transition

The study of the dynamics of the glass transition was again a strong activity of the college. The studies included samples of very different types (ionic and electrolyte glasses, low molecular weight organic liquids, polymers, etc.) which is important as the universality of the glass transition is not proofed up to now. From the view of inelastic neutron scattering, however, the experimental observations are quite similar for most of the samples. Generally speaking, the elastic scattering (i.e. relaxation slower than  $\sim 10^{-9}$  sec) is decreasing rapidly with increasing temperature around the macroscopic glass transition temperature  $T_g$ , apparently associated with the appearance of a fast ( $\sim 10^{-12}$ sec), local ( $< \sim 1\text{\AA}$ ) relaxational process. The width of the observed corresponding quasielastic scattering peak seems to be independent of Q and only weakly temperature dependent, whereas the intensity increases rapidly with temperature. The interpretation of this fast component is controversial up to now, ranging from changes in the density of states of the phonons to fast local relaxations. Indeed it was shown for polyethylene in the melt [14] that neutron scattering does not allow to decide whether this fast motion is of the diffusional or vibrational type, as the observed quasielastic scattering can be interpreted satisfactorily by both models. Low temperature measurements on drawn deuterated PE are in favour of the second possibility. This is consistent with the results from polybutadiene ( $(\text{C}_4\text{H}_6)_n$ ) where the mean squared displacements at low temperatures ( $< T_g - 30\text{K}$ ) show a behaviour as expected from an harmonic oscillator [15] but

a pronounced increase near  $T_g$  (see Fig. 57). Analysing the observed scattering intensity at low energy transfers, this can be interpreted as a change in the phonon density of states [16] (see Fig. 58).

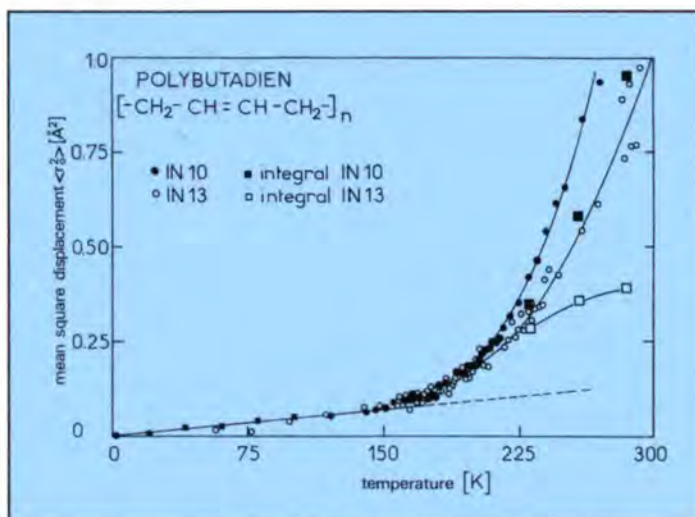


Figure 57: Mean squared displacements in polybutadiene as a function of temperature [15] measured with different elastic resolution.

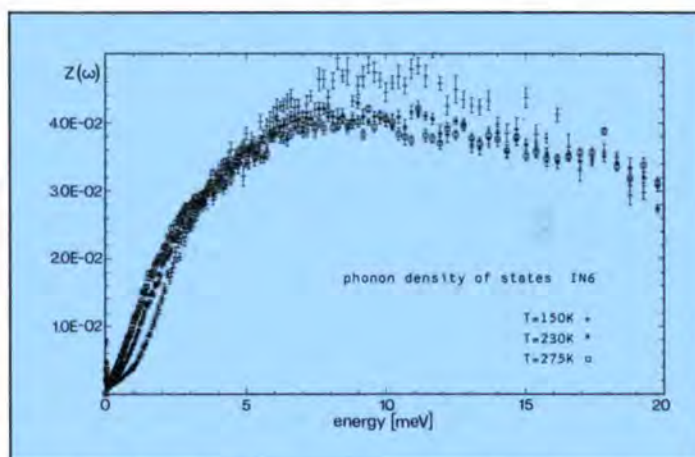


Figure 58: Changes in the phonon density of states in polybutadiene as function of temperature near the glass temperature ( $T_g \sim 186\text{K}$ ) [16].

Evaluation of measurements (IN6) on the electrolyte glass  $\text{BeCl}_2 \cdot 6.7\text{D}_2\text{O}$  [21] in terms of amplitude weighted density of states  $P(E)$  show again a change of  $P(E)$  in the energy range  $0.4\text{meV} < E < 1.5\text{meV}$  arising near the glass transition temperature. The relation of these dynamic effects to structural rearrangements was studied also on D20 [21], where in fact a change in the first peak of the static structure factor was found near  $T_g$ .

Several of the neutron scattering results are compared to mode coupling theories [17] predicting a square root temperature dependence for the elastic scattering (for structural relaxation) and scaling laws for the time dependence of the structural as well as secondary relaxations. The predicted square-root temperature dependence of the formfactor corresponds roughly to the observations made in polybutadiene [15] and trinaphthylbenzene ( $\text{C}_{34}\text{H}_{26}$ ) [18].

Concerning the scaling laws for secondary relaxations the observed quasielastic scattering) was found to be not a simple Lorentzian for trinaphthylbenzene [18], ii) its energy dependence

was found to correspond to the scaling factor predicted for the secondary relaxation for polybutadiene [15] and iii) careful studies of the intensity decrease with energy transfer (IN13) on  $\text{Ca}(\text{NO}_3) \times \text{Na}(\text{NO}_3)$  [19] show a scaling parameter consistent with the predictions for the secondary relaxations by the mode coupling theory. Studying the structural relaxation in  $\text{Ca}_{0.4}\text{K}_{0.6}(\text{NO}_3)_{1.4}$  [20] spin echo measurements had shown up the non exponential relaxation as well as a non-ergodic fraction of  $\sim 84\%$  of the frozen structure (see annual report 86) and this could be confirmed for further Q-values.

In spite of these results none of the experiments was able to prove up to now the validity of the mode coupling theory. This is mainly due to the complexity of the phenomena involving quite different time and local scales, and the difficulty to separate phonon contributions. Thus the study of the glass transition will require further experiments on various types of instruments and on different kinds of samples.

## Metal Physics

The self-diffusion of the IVb metals Ti, Zr and Hf, which undergo a martensitic phase transformation from the high temperature bcc-phase ( $\beta$ -phase) to a hcp-structure ( $\alpha$ -phase), is very fast with respect to other bcc metals and its diffusivities show remarkable deviations from an Arrhenius law. Furthermore, solute atoms like Co and Fe diffuse orders of magnitudes faster than the corresponding self diffusion.

Whereas QNS studies on bcc- $\beta$ -Ti single crystals showed that the self-diffusion takes place mainly via  $\langle 111 \rangle$  nearest neighbour vacancy jumps (report 86), the mechanism in alloyed samples is still unresolved. As a first step to understand the local distortion of the host matrix around a solute atom lattice parameter measurements were performed on pure  $\beta$ -Zr and  $\beta$ -ZrCo(2%Co) in the high temperature  $\beta$ -phase on D2B (see Fig. 59) [29]. Samples of pure Zr and Zr-2 at %Co were sealed into vacuum tight Nb-containers to prevent the getting of impurity atoms like oxygen at high temperatures. The fact that the lattice parameter for the alloy is lower than for pure Zr (Co has a larger atomic radius than Zr) indicates that the solute atoms occupy mainly substitutional sites. From Fig. 59 it can also be deduced that lattice distortions due to the alloying increase, when the temperature is lowered towards the martensitic phase transition at  $860^\circ\text{C}$ .

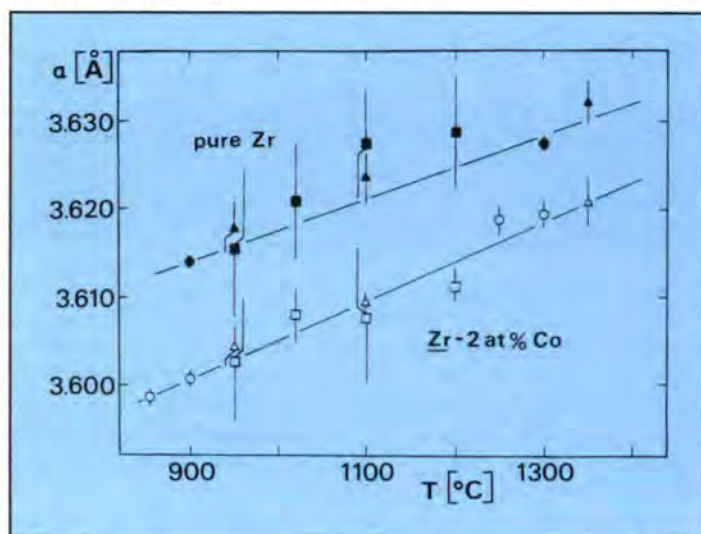


Figure 59: Lattice parameters of pure and alloyed Zirconium as measured on D2B (Heiming, Trampenau, Petry, ILL report 6-12-100).

Alloying Nb to Zr normalizes the  $\beta$ -Zr self-diffusion, which is very similar to that of  $\beta$ -Ti, in two respects. Scaled to the melting temperature the diffusivity decreases and its temperature dependence approaches an Arrhenius law. Following the idea that the phonon softening is mainly responsible for the anomalous diffusion behaviour of bcc metals, the soft phonon should become less pronounced with increasing Nb content in ZrNb alloys. First measurements of the LA  $\langle 111 \rangle$  phonon branch confirm this assumption and show that at the corresponding  $q = 2/3 \langle 111 \rangle$  value not only a softening appears but also a strong diffuse elastic scattering (roughly 10-times the inelastic intensity) [29]. Phonon intensity at this “ $\omega$ -point” is found at almost zero energy transfer. Upon changing the Nb content from 16 at% to 28 at% the minimum phonon frequency is shifted towards a higher value, while the rest of the LA  $\langle 111 \rangle$  phonon curve stays constant within the experimental error. Further measurements on different alloy compositions were able to show a quantitative relationship between the phonon softening and the change of migration enthalpy in the ZrNb system. It is important to mention that the measurements were performed at  $800^\circ\text{C}$ , i.e. within thermal equilibrium of the high temperature  $\beta$ -phase. Apparently the soft LA  $2/3 \langle 111 \rangle$  phonon leads to a stable distortion towards the  $\omega$ -phase (metastable phase, usually observed after quenching) already at temperatures well above the  $\alpha$ - $\beta$  transition.

## Fast Ionic Conductors

The study of  $\text{Cu}^+$  cation diffusion in glassy  $(\text{CuPO}_3)_{0.5}(\text{CuI})_{0.5}$ , a fast ionic conductor, revealed a much lower activation energy than previously observed for  $\text{Ag}^+$  glasses [22]. These measurements were carried out on IN6 ( $\lambda_i = 5.1\text{Å}$ , resolution  $80\ \mu\text{eV}$  FWHM,  $1.44\text{Å}^{-1} < Q < 2.04\text{Å}^{-1}$ ) and the spectra consist of a quasielastic broadening of the elastic line and an inelastic tail up to 20 meV. The deconvolution of elastic and quasielastic contributions gives a constant value for the purely elastic contribution. The diffusion coefficient obeys an Arrhenius law with  $D = 2.75 \times 10^{-5} \exp[-E_a/RT] \text{cm}^2\text{s}^{-1}$  with  $E_a = 2.6\text{kJ mol}^{-1}$ . The motion described here occurs certainly on a local scale without contributing to a long range diffusion which is involved in ionic conductivity.

Quasielastic incoherent scattering experiments on a single crystal of  $\text{Na}^+\beta\text{Al}_2\text{O}_3$  studied the  $\text{Na}^+$  motion for Q-values parallel to the sample conduction plan between  $25^\circ\text{C}$  and  $400^\circ\text{C}$  (IN6:  $4.1\text{Å}$ ,  $5.1\text{Å}$ ). The data are interpreted within a model where only a fraction of  $\text{Na}^+$ -ions are mobile on a picosecond scale. About 18% of  $\text{Na}^+$  ions, corresponding to doubly occupied cells, undergo a restricted localized motion with a residence time of about  $3 \times 10^{-11}$  s and about 63% of ions undergo long range motions on a time scale of  $1.5 \times 10^{-10}$  s. The conductivity mechanism can be understood as a kind of caterpillar mechanism combined with an itinerant polaron model [23].

## Hydrogen in Metals

Recent theoretical considerations suggest that at low temperatures the transport properties of light interstitials in metals are dominated by the interaction with the conduction electrons. Their influence results in two main effects: (i) the screening electron cloud which accompanies the light particle reduces the tunneling matrix element J between adjacent sites by a factor proportional to power law in T; (ii) electron scattering causes damping of the particle motion which is proportional to T. At low enough temperatures electron scattering

dominates and a diffusion coefficient that increases with decreasing temperature is predicted:  $D \sim T^{2K-1}$ , where  $K$  is an electron coupling coefficient ( $K < 0.5$ ). Finally below a crossover temperature  $T^*$  the hopping changes to coherent tunneling. As it appears by now for hydrogen the regime of electron assisted hopping can only be accessed on systems where the hydrogen is trapped at an impurity and thus kept in solution at low temperatures. Quasielastic neutron scattering experiments were performed on  $NbN_xH_y$  ( $x = 0.004$ ,  $y = 0.003$ ) using IN6 [24]. The observed spectra are composed of elastic and quasielastic lines. Fig. 60 shows the weight and width of the quasielastic component as function of temperature. Starting from a plateau at low temperature the effective linewidth decreases with increasing temperature and reaches another plateau above 100K. At the same time the weight of the quasielastic component or the number of hopping protons increases greatly. Qualitatively the decrease of the quasielastic linewidth with increasing temperature shows the predicted effect. Contrary to what is expected in thermally activated processes the H-motion slows down with increasing temperature revealing its quantum nature. At higher temperatures the onset of phonon activation may counterbalance the electronic effects and lead to the observed plateau in the linewidth.

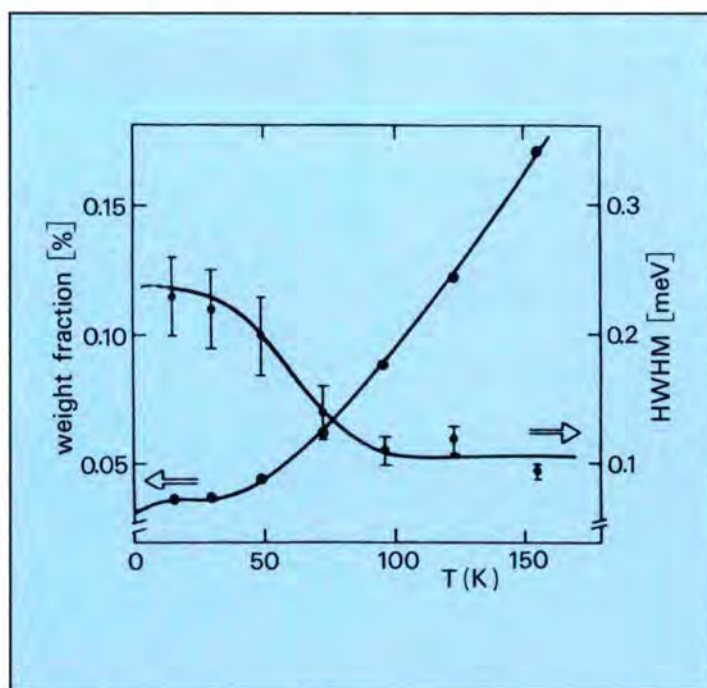


Figure 60: Quasielastic linewidth and weight of the quasielastic component as observed in  $NbN_{0.004}H_{0.003}$  as a function of temperature [24].

Hydrogen diffusion in amorphous alloys is more complex in comparison to diffusion in single- or polycrystals. Whereas for the latter in general all sites occupied by hydrogen are equivalent, or are well defined at least (like octahedral and tetrahedral sites in hexagonal host lattices), in amorphous alloys a whole spectrum of sites is expected due to chemical or/and structural disorder. This corresponds to a distribution of site energies and saddle point energies. By macroscopic methods information on the distribution of site energies can be obtained by measuring the free partial energy of hydrogen dissolution in amorphous metals as a function of hydrogen concentration. In the case of amorphous Pd-Si (a metal-non metal glass) one Gaussian distribution is appropriate to

describe the H-solubility from a few ppm to 0.7 H/Pd, while QNS data cannot be understood in such a model. In a metal-metal glass as amorphous  $Ni_{35}Ti_{65}$  more than one Gaussian distribution appears already to be necessary to understand hydrogen solubility and to account for the different combinations of Ti and Ni atoms on the corners of a tetrahedron. Up to now quasielastic neutron scattering has revealed very detailed information on microscopic aspects of H-diffusion and recent measurements on IN10 [25] have shown that the amorphous Ni-Ti system is especially suitable for studying hydrogen motion by QNS. The observed quasielastic peak could not be fitted with one Lorentzian and there are at least two types of jump processes within the energy range accessible on IN10. In addition, the integrated area is less than unity at high wavevectors. Therefore, it is assumed that there is a third fast process outside the energy range of IN10 which needs to be followed up on an instrument with coarser energy resolution but wider dynamical range.

The diffusion of hydrogen or deuterium at higher concentrations in metals involves contributions from collective diffusion (seen by coherent scattering) which can be separated exactly from incoherent contributions only by polarization analysis. First measurements [26] on 70% D in Nb at high temperature (600K) on D7 with an elastic resolution of 150  $\mu\text{eV}$  (HWHM) are shown in Fig. 61. The data convincingly show the separation of coherent and incoherent scattering which would be difficult by other methods due to similar linewidths of these contributions. While the intensity of the coherent scattering is increasing with increasing  $Q$  due to short range order effects, as expected the incoherent intensity stays roughly constant with  $Q$ . In general the coherent widths are broader than their incoherent counterparts revealing the retardation of tracer diffusion due to blocking in local correlations. These results were achieved in a - for polarization analysis - relatively short measuring time ( $2 \times 40$  hours), the supermirror transmission being high for low energy transfers.

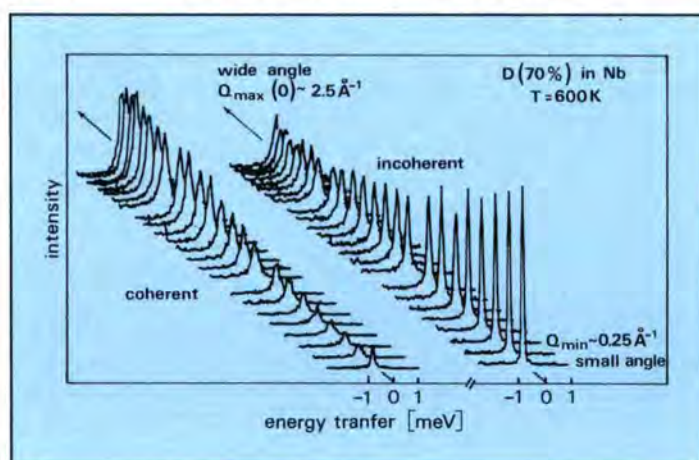


Figure 61: Self- and distinct part of the dynamical scattering law for 70% deuterium in Nb as measured by polarization analysis on D7 [26].

## Gelation of Alumino-Silicates

The structure of gels made by hydrolysis and polycondensation of alkoxides could be more readily controlled if the two stages could be separated in time, but since hydrolysis is slow, and condensation which is responsible for the structure is fast, the two processes become mixed.

An alternative approach is to incorporate two stage hydrolysis using a mixed alkoxide  $R'O-M'-O-M'-OR'_3$  offering different reactivity for the two end members. SANS and NMR studies of the kinetic gelation of  $(BuO)_2Al-O-Si(OEt)_3$  shows a rapid hydrolysis of the  $Al-(OBU)_2$  groups to create a sol of dense particles with  $Al-O-Al$  linkages surrounded by organic  $Si-OR$  groups which prevent extension of the particles. The polymerisation reaction then requires hydrolysis of the siloxane members - a process which is slow and directly related to the available water. The degree of control inherent on the process is shown by the Guinier radius, which is less than  $30 \text{ \AA}$  after the initial  $Al-OR$  hydrolysis and climbs to  $100 \text{ \AA}$  in 30 hours at a rate depending on the free water concentration. This slow, controlled growth leads to a rather regular gel structure as derived from the interference maximum in the scattering curve, which can be fitted to a simple hard sphere extended "excluded volume" model.

## Applied Science

Extensive investigations of the residual stress field around the tip of fatigue cracks have shown that the residual stresses play a decisive role for the crack growth and hence the lifetime of technical components. Samples of different thickness ( $3-20\text{mm}$ ) have been studied on DIA in order to explore in how far surface sensitive X-ray results are representative for the bulk. Whereas for thin samples X-ray and neutron results agree in finding a planar stress field, for thick samples the interior stress field differs considerably from that at the surface. At the crack tip the differences can be well characterized by a compressive hydrostatic pressure which has to be added to the stress values observed at the surface to give the stress value in the interior [27].

A further more exotic subject was a QNS study (IN5) of the mobility and the fraction of the free pore water in different cements and how this is related to the bound water present. The experiments resulted in determining the temperature dependence of the self diffusion coefficients for the free water fraction and showed that the diffusion constants are considerably lower than for free water. First studies suggest that the rotational bond length is about  $2\text{\AA}$  as compared to only  $0.9\text{\AA}$  in bulk water. [28].

Secretary: B. Frick

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### Quasicrystals and six-dimensional crystallography

The term “quasicrystal” is short for “quasiperiodic crystal”. It describes a new class of incommensurate crystal structures which have  $\delta$ -functions in their Fourier transform but which (in contrast to incommensurately modulated crystals) have point symmetries incompatible with periodic order. This is abhorrent to the intuitions of crystallographers, but manifestly possible: the Penrose tiling is a geometrical structure, known for a decade, with five-fold symmetries and Bragg diffraction.

What are the actual atomic arrangements of atoms in the real icosahedral alloys, and how are these related to the diffraction pattern? Merely proposing a structural model requires developing a scheme to represent the model structure by a finite number of parameters. This is intrinsically more difficult than for a crystal. A perfect quasiperiodic quasicrystal without disorder still has an infinite number of sites which are not exactly equivalent, since the structure is aperiodic.

A straightforward approach is to consider the density of atoms to be a cut through a truly periodic density in a higher D-dimensional space. This is valid for all kinds of incommensurate crystals - both quasicrystals and ordinary crystals with incommensurate modulations.

Let the D-dimensional space be for instance a six-dimensional space which can be spanned by six orthonormal vectors  $\underline{e}_i, i = 1-6$ . These vectors can be identified as the rows of the unit  $6 \times 6$  matrix

$$\underline{I} = \begin{vmatrix} 1 & 0 & 0 & 0 & 0 & 0 \\ 0 & 1 & 0 & 0 & 0 & 0 \\ 0 & 0 & 1 & 0 & 0 & 0 \\ 0 & 0 & 0 & 1 & 0 & 0 \\ 0 & 0 & 0 & 0 & 1 & 0 \\ 0 & 0 & 0 & 0 & 0 & 1 \end{vmatrix} = \begin{vmatrix} \underline{e}_1 \\ \underline{e}_2 \\ \underline{e}_3 \\ \underline{e}_4 \\ \underline{e}_5 \\ \underline{e}_6 \end{vmatrix}$$

Within this six-dimensional space, it is possible to further define two spaces V and W spanned by vectors which can be identified as the row vectors of the two matrices

$$\underline{V} = \begin{vmatrix} \sqrt{5} & 1 & -1 & -1 & 1 & 1 \\ 1 & \sqrt{5} & 1 & -1 & -1 & 1 \\ -1 & 1 & \sqrt{5} & 1 & -1 & 1 \\ -1 & -1 & 1 & \sqrt{5} & 1 & 1 \\ 1 & -1 & -1 & 1 & \sqrt{5} & 1 \\ 1 & 1 & 1 & 1 & 1 & \sqrt{5} \end{vmatrix} = \begin{vmatrix} \underline{v}_1 \\ \underline{v}_2 \\ \underline{v}_3 \\ \underline{v}_4 \\ \underline{v}_5 \\ \underline{v}_6 \end{vmatrix} \quad (1)$$

and

$$\underline{W} = \begin{vmatrix} -\sqrt{5} & 1 & -1 & -1 & 1 & 1 \\ 1 & -\sqrt{5} & 1 & -1 & -1 & 1 \\ -1 & 1 & -\sqrt{5} & 1 & -1 & 1 \\ -1 & -1 & 1 & -\sqrt{5} & 1 & 1 \\ 1 & -1 & -1 & 1 & -\sqrt{5} & 1 \\ 1 & 1 & 1 & 1 & 1 & -\sqrt{5} \end{vmatrix} = \begin{vmatrix} \underline{w}_1 \\ \underline{w}_2 \\ \underline{w}_3 \\ \underline{w}_4 \\ \underline{w}_5 \\ \underline{w}_6 \end{vmatrix} \quad (2)$$

Each of these matrices is of rank three, i.e. only three of the six vectors defined in each space are required to span the space. Moreover, these two three-dimensional spaces within the six-dimensional space are orthogonal, since

$$\underline{V} \underline{W} = \underline{W} \underline{V} = 0.$$

Within each space, the angle between the  $i^{\text{th}}$  row vector and the  $k^{\text{th}}$  row vector is  $\pm \arccos(1/\sqrt{5})$ , so that the row vectors can be identified with vectors defining the vertices of icosahedra, as in figure 62. Each basis vector  $\underline{e}_i$  in the six-dimensional space can now be written as

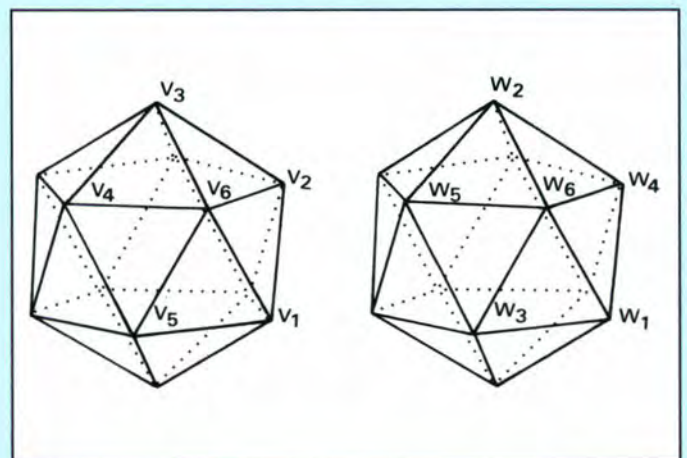


Figure 62: Icosahedra at the origin showing the relative orientation of the row vectors  $\underline{v}_i$  and  $\underline{w}_i, i = 1-6$  spanning the two spaces V and W. The vectors are represented as vertices of these icosahedra.

$$\underline{e}_i = \frac{1}{2\sqrt{5}} (\underline{v}_i - \underline{w}_i)$$

and hence the  $\underline{e}_i$  can be resolved into components in, or equivalently projection onto the two spaces. The projection onto W space of a vector  $\underline{e}_i$  may be defined as its elevation  $E(\underline{e}_i)$  and the projection onto V space as its plan  $P(\underline{e}_i)$ .

Consider now the projection onto W space of a six-dimensional cube centred at  $[0,0,0,0,0,0]$  and with vertices at  $[\pm 1/2, \pm 1/2, \pm 1/2, \pm 1/2, \pm 1/2]$ . The projection of this cube is defined by the shape formed by the elevations

of each of its 64 vertices. These elevations can be sorted into four distinct groups according to their size.

The twelve elevations with the largest moduli of  $\tau/\sqrt{2}$  are defined by

$$\pm \frac{\tau}{2\sqrt{5}} \underline{W}_i \quad i = 1-6 \quad (3a)$$

where  $\tau = (\sqrt{5} + 1)/2$ . One example of such elevation is

$$E \left( \frac{1}{2}, \frac{1}{2}, \frac{1}{2}, \frac{1}{2}, \frac{1}{2}, -\frac{1}{2} \right) = \frac{\tau}{2\sqrt{5}} \underline{W}_6. \quad (3b)$$

The 20 next largest have moduli of  $[0.3(\tau + 2)]^{1/2}$ , such as

$$E \left( \frac{1}{2}, \frac{1}{2}, \frac{1}{2}, \frac{1}{2}, -\frac{1}{2}, -\frac{1}{2} \right) = \frac{\tau}{2\sqrt{5}} [\tau^2, \tau^{-1}, \tau^{-1}, \tau^2, \tau^{-1}, -\tau]. \quad (4)$$

These two groups of elevations can be identified as defining the vertices at the fivefold and threefold axes respectively of a rhombic triacontahedron in  $W$  space centred at the origin. The remaining two groups of elevations divide into a group of 20 with moduli  $\tau^{-1}[0.3(\tau + 2)]^{1/2}$  and a group of 12 with moduli  $\tau^{-1}/\sqrt{2}$ . These two groups of elevations are vectors proportional to the threefold and fivefold axis vectors of the rhombic triacontahedron defined by the first groups of elevations, but lie within this rhombic triacontahedron.

Thus the projection of a six-dimensional cube onto  $W$ , i.e. its elevation, is a rhombic triacontahedron. For the cube centred at  $[0,0,0,0,0,0]$ , the face centres of this rhombic triacontahedron are then expressible as the 15 vectors

$$\underline{h} = \pm \frac{1}{2} \frac{\tau}{2\sqrt{5}} (\underline{W}_i \pm \underline{W}_j) \quad i \neq j \quad i, j = 1-6 \quad (5)$$

and are vectors such as

$$\begin{aligned} \frac{\tau}{4\sqrt{5}} (\underline{W}_1 - \underline{W}_2) &= \frac{\tau}{2\sqrt{5}} [-\tau, \tau, -1, 0, 1, 0] \\ \frac{\tau}{4\sqrt{5}} (\underline{W}_1 + \underline{W}_3) &= \frac{\tau}{2\sqrt{5}} [-\tau, 1, -\tau, 0, 0, 1] \\ \frac{\tau}{4\sqrt{5}} (\underline{W}_1 - \underline{W}_6) &= \frac{\tau}{2\sqrt{5}} [-\tau, 0, -1, -1, 0, \tau] \end{aligned} \quad (6)$$

It follows that the condition for the elevation  $\underline{Y}$  of a particular point  $\underline{X} = [a, b, c, d, e, f]$  of the six-dimensional space to lie within this rhombic triacontahedron  $R$  is simply

$$|\underline{X} \cdot \underline{h}| \leq \underline{h} \cdot \underline{h} \quad (7)$$

or equivalently

$$|\underline{X} \cdot \underline{h}| < 0.1 (\tau + 1) (\tau + 2) \quad (8)$$

for each  $\underline{h}$  defined by equation (5). The full set of 15 inequalities is obtained by cyclically rotating the first five coordinates of each of the three expressions on the right-hand side in equation (6).

A three-dimensional quasiperiodic lattice may now be defined by stating that its vertices are the plans of those 6D-cubes whose elevations lie within  $R$ . The conditions for this to hold are that inequalities in equation (8) is satisfied.

The method is illustrated in figure 63 for the simplified case of a 1D-quasiperiodic lattice generated from a 2D square lattice by strip-projection.

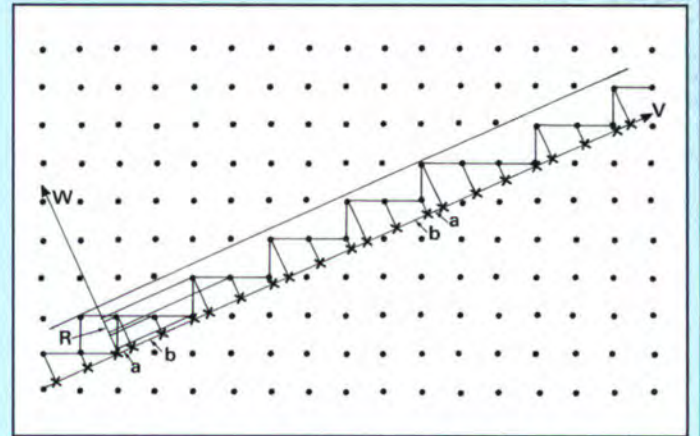


Figure 63: Illustration of the strip-projection method showing a 1D quasiperiodic structure (x) generated from a 2D square lattice (\*).

In principle, a 6-dimensional density can be reconstructed, given the complex amplitudes of all the Bragg spots in real 3-dimensional space, by assigning them to the appropriate 6-dimensional wavevectors and then Fourier transforming back into 6-dimensional real space.

The whole apparatus of crystallography is now applicable in the 6-dimensional space. There are 16 icosahedral space groups in 6-dimensions. The known icosahedral quasicrystals correspond to a  $D=6$  simple cubic structure with inversion (m 35).

There is no conceptual obstacle to adapting the direct methods of phase determination known to crystallographers and pursuing a structural determination in  $D = 6$  by brute force. Unfortunately, this approach may be impracticable for understanding the microscopic structure because of the too many parameters to be determined.

Furthermore, the Bragg part (not the diffuse part) of the scattering is encoded by the periodic components of the density only, and therefore gives a mere "averaged" structure in real space. Nonetheless, the motion of cut through a higher dimensional density and projection into the physical space, includes and unifies the cases of non periodic space tiling and density waves and might appear as very useful for the understanding of particular problems in classical crystallography. Extra extinctions attributed to atomic order in the unit cell, relation between different structures in 3D or description of the structure of complex phases (Laves phases, Frank-Kasper phases...) are examples of problems that can be solved by treating them from rational cut in proper hyperspaces.

# COLLEGE 8 AND EMBL OUTSTATION BIOCHEMISTRY

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## Introduction

The majority of biological proposals continues to involve low resolution structural studies on the small angle scattering instruments D11 and D17, both of which have worked without incident throughout the year. D16 has continued to provide a wealth of data particularly on membrane systems whilst DB21, the joint ILL/EMBL diffractometer has been used continuously for low resolution crystallographic studies. High resolution work on D19 has been extended from protein crystallography to fibre diffraction on DNA.

## Scientific Trends and Highlights in 1987

### Solution scattering

#### Protein/nucleic acid interactions

The natural contrast for neutrons between nucleic acids and proteins facilitates the study of a wide range of nucleoprotein complexes. In particular, several projects are under way studying various components of the gene regulation, transcription-translation pathway.

One of the major long-term projects is the study of DNA-dependent RNA polymerase (RNAP). RNAP catalyzes the transcription of genetic information from its storage form (DNA) into the "blue-print" form messenger RNA that is used for translation into proteins by the ribosomes.

Initially, the arrangement of the subunits,  $\alpha_2\beta\beta\sigma$ , had been studied in the isolated protein. The interest is now turning towards the investigation of the active complexes of RNAP with DNA and RNA. By means of genetic engineering, a well-defined short DNA fragment has been produced in protonated and deuterated form. By specific deuteration of the single parts of the complex (DNA, the whole RNAP or single subunits) and by using the contrast matching technique with  $D_2O$ , it was found that the long axes of the roughly triangular RNAP molecule and of DNA run parallel with an axial separation of 5 nm (MPI Martinsried and ILL). Fig. 64

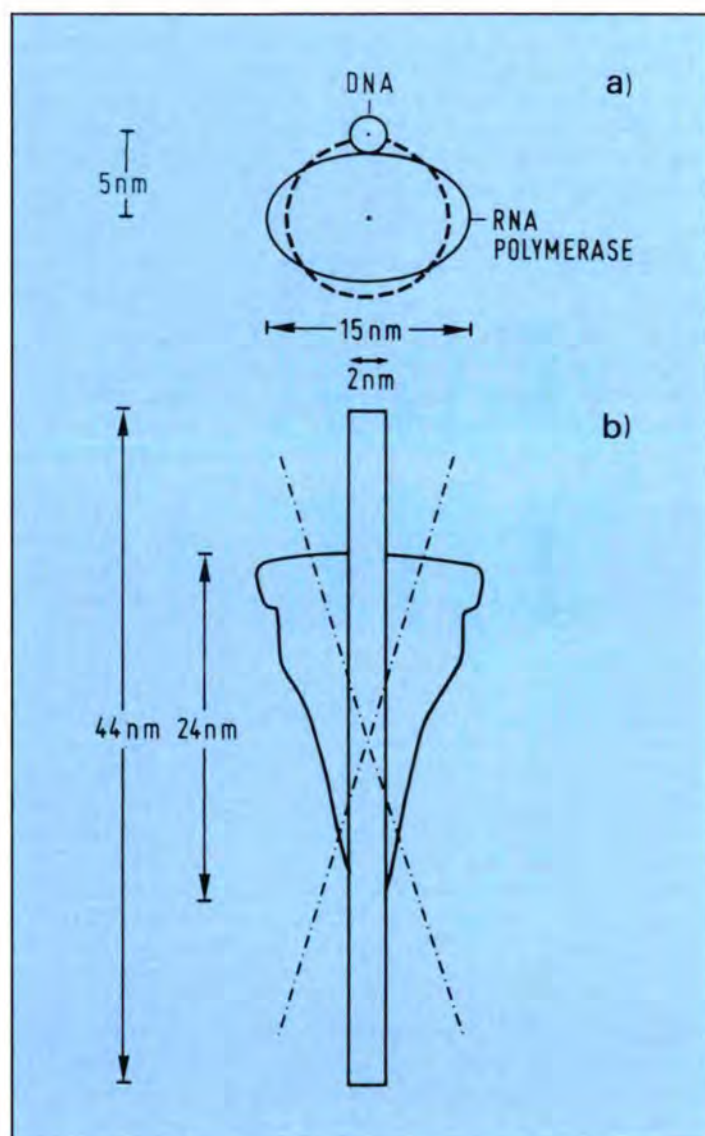


Figure 64: Model of the RNA polymerase-promoter complex. (a) View along long axes of RNAP and DNA. RNAP is represented with circular (---) and elliptical cross-section (—). (b) Top view of the complex.

Other important DNA-protein complexes are that of DNA and repressors regulating transcription and thus the production of certain proteins. The tet repressor controlling the resistance of bacteria to the antibiotic tetracycline is being studied with neutron small-angle scattering. The tet repressor is a dimeric molecule; two repressors are bound to a tandem of operators

on the DNA separated by 30 base pairs. A first result of the scattering experiments is a distance between the repressors' centres of mass of 11 nm. From the high quality of the data we can expect that more details concerning the orientation of the molecules, and on whether or not DNA is bent at the operators can be obtained (MPI Martinsried and ILL).

RecA protein, which *in vitro* is able to promote strand exchange between two homologous DNA molecules, is being extensively studied in filamentous complexes with DNA (ILL, IBMC Strasbourg). Through contrast variation and using perdeuterated DNA it has been shown that DNA lies on the axis of the complex of the protein with ds DNA. Data have been obtained on a variety of complexes of recA with single-stranded and double-stranded DNA as well as self polymers of the protein revealing structural differences between them. Preliminary experiments aimed at orienting the complexes in a shear gradient have proved successful and lead to the hope that even more detailed information on the helical structure of the complexes may be obtained.

### **E. coli large ribosomal subunit**

The ribosomes, particles composed of two large and one small RNA molecules and more than 50 different proteins and organized in two subunits of a mass ratio of 2:1, synthesize new proteins on their surface using the code contained in the messenger RNAs. For a number of years, small-angle scattering measurements (Max-Planck Institut, Berlin and ILL) have been devoted to the study of the arrangement of the proteins within the large (50S) subunit. In 1987, the study has been extended to include a first set of protein pairs in the 70S particle (the whole ribosome). The distances between proteins in different subunits will finally allow location of the two subunits unequivocally with respect to each other. The currently available data set is not yet sufficient for this purpose; it may only give hints concerning the mutual orientation. A few measurements concerned pairs within the small (30S) subunit. Such data are needed to check the agreement of our data with those of the group of Moore *et al.*, which will provide an anchor for fixing the inter-subunit arrangement. As in earlier years, the main part of the investigations was directed towards filling the 50S distance table with new measurements of protein pairs. The results on the large subunit processed so far comprise more than 40 distance measurements and 22 *in situ* shape determinations. Several data sets have been obtained repeatedly. A spatial distribution of the mass centres of about 16 proteins will result from a current treatment of the whole data set with a new procedure. It consists in fitting the Fourier transform of up to six, in general two to three "moving" B-splines of varying location, width and height representing the intra- and inter-protein vectors in real space to the scattering data. This procedure has the advantage of requiring a minimal number of physically meaningful parameters. The method has been tested on model scattering curves and published data.

### **Membrane proteins**

The advances of recent years in the purification of membrane proteins by solubilisation in detergent has led to several such systems being studied by neutron small angle scattering. Contrast variation has allowed the determination of the *in situ* molecular weight of several reaction centres and light harvesting complexes in detergent micelles (Freiburg, ILL).

### **Protein stabilization in halophiles**

Proteins in halophilic bacteria are in a saturated KCl environment, which would normally inhibit structure and function in the better known eubacterial and eukaryotic systems. They are interesting illustrations of processes that have evolved to cope with the fundamental problem of protein folding in a given environment to yield a stable and functional structure. For a few years now, these processes have been studied by small angle neutron scattering (Weizmann Institute, ILL), through experiments on the solution structure and solvent interactions of malate dehydrogenase from *Halobacterium maris mortui*. Data on the protein in  $MgCl_2$  containing solvents had shown that, unlike other salt conditions studied so far, the protein was stable only at intermediate concentrations of this salt. The results on the solution structure and interactions from neutron experiments in similar conditions have led to the proposal of a model for the stabilization of the protein in its high salt environment which involves the formation of a protein-salt hydrate defined by the tertiary structure of the protein and the nature and hydration properties of the salt. Protein-nucleic acid interactions in halophiles also pose problems of fundamental importance, because of the saturated salt environment that, normally, is a strong inhibitor of such interactions. A halophilic elongation factor Tu has been characterised by neutron solution scattering in a first step towards studying its interactions in high salt solvents with aminoacyl-tRNA molecules (EMBL, ILL).

### **Crystallography and fibre diffraction**

The high resolution diffractometer D19 has been successfully used both for protein crystallography and, for the first time, for fibre diffraction studies of DNA.

### **Protein-solvent interactions**

Solute/solvent interaction studies have continued with measurements on hen egg-white lysozyme soaked in tetramethylammonium hydrochloride (TMACl) mixtures (Birkbeck College, ILL), and dimethylsulfoxide (DMSO). Using completely deuterated solute molecules and  $H_2O$  mother liquor the signal from the solute is large, so that even partly occupied sites and disordered molecules can be detected. For lysozyme interacting with TMACl data were recorded for 18% and 30% (w/w) of TMACl ( $D_{12}$ ) in  $H_2O$ . For both crystals there was a noticeable reduction in unit cell volume compared to native lysozyme, which led to some reduction in scattering power at high angle, and data were only recorded to 2.4 Å giving 3900 reflections. The change in structure was however sufficiently small to allow analysis to start from the native structure, and a detailed study is now in progress.

For the lysozyme-DMSO analysis the solution held 15% DMSO ( $D_6$ ) (V/V). In this case data were recorded to 1.75 Å resolution giving 10200 reflections. The DMSO molecule has a very large dipole moment of 4 Debye, which should ensure reasonable fixation of the molecules, and at the present stage of the analysis 9 molecules have been found. Data with normal DMSO in  $D_2O$  mother liquor have also been recorded ( $d = 2$  Å), and they will eventually be used as cross-check; for each positive DMSO ( $D_6$ ) peak in the  $H_2O$  solvent we should find a negative DMSO ( $H_6$ ) peak in the  $D_2O$  solvent. This last set of data, giving 5900 reflections was measured in five and a half days, showing that the new D19 allows rapid data collection for a small protein if mm large crystals are available.

## Fibre diffraction

The fibre diffraction study on the synthetic polynucleotide poly d(A-T) d(A-T) was also carried out to determine the positions of water molecules around the DNA double helix. Fig. 65 shows the scattering pattern recorded on the D19 multidetector from the highly ordered fibres. Data were recorded to 3 Å resolution from fibres immersed both in H<sub>2</sub>O and D<sub>2</sub>O. Using the model of the DNA derived from X-ray diffraction a Fourier difference synthesis between the H<sub>2</sub>O and D<sub>2</sub>O immersed structures was calculated. The resulting isotopic difference map, of which a projection is shown in Fig. 66, has been interpreted in terms of the water molecule locations around the DNA.

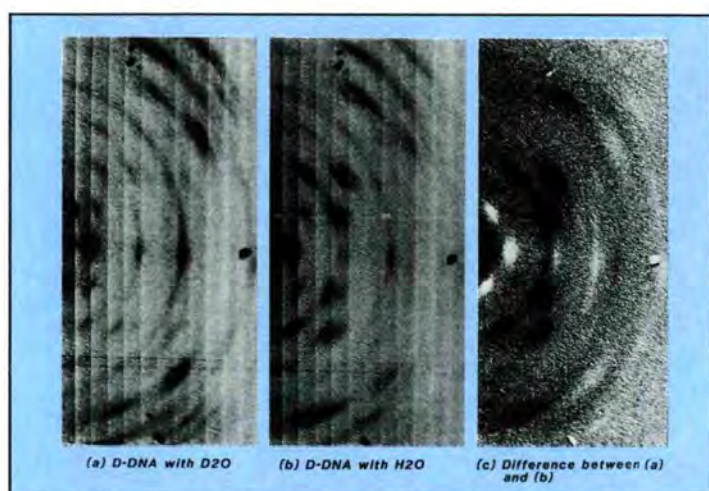


Figure 65: Neutron diffraction data: (a) D-DNA with D<sub>2</sub>O; (b) D-DNA with H<sub>2</sub>O and (c) difference between images in (a) and (b).

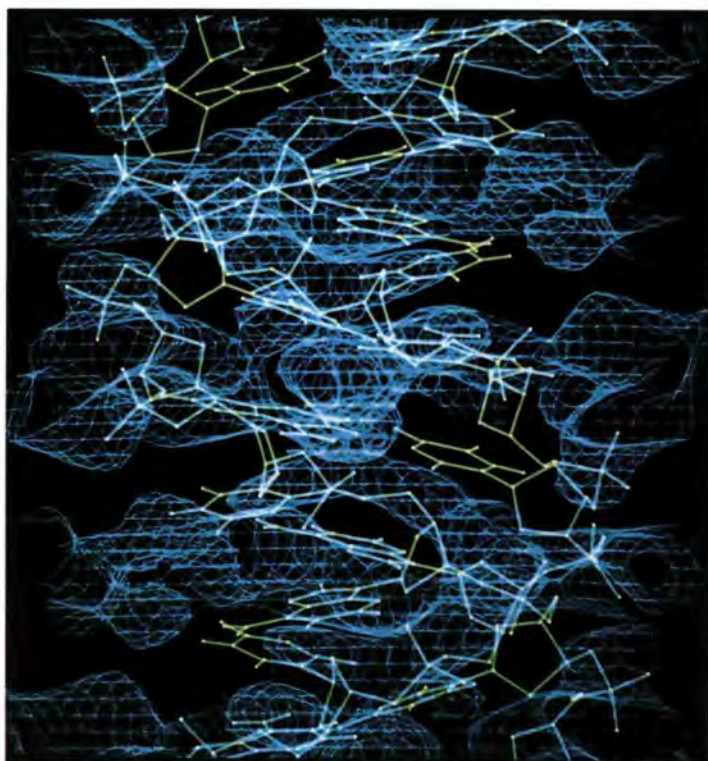


Figure 66: Projection along the b-axis of the D-DNA unit cell, showing a skeletal representation of the D-helix (green) and difference density (blue) associated with water around the structure.

Studies have also been carried out on D11 and D17 of superhelical forms of DNA (ILL and EMBL). The ubiquitous distribution of superhelical DNA calls for detailed knowledge of its conformation. There is evidence for the existence of two distinct structures for supercoiled circular duplex DNA: in the toroidal form the DNA is wound into a circular solenoid while in the interwound form two duplex chains intertwine into an extended coiled-coil. It has been shown that solutions of superhelical DNA can in certain conditions spontaneously form an ordered liquid-crystalline phase which is susceptible to magnetic orientation. Neutron diffraction shows the presence of a two-dimensional hexagonal lattice (Fig. 67) which is typical of stiff or semi-flexible rod-like polyelectrolytes (cf. TMV gels, Pf1 and fd filamentous phages) and is therefore strong evidence for an interwound form. Experiments are being carried out to observe the superhelical pitch directly with neutron diffraction on oriented liquid-crystals and with X-ray diffraction of partly hydrated fibres. Neutrons unlike X-rays do not cause nicking with the consequent destruction of the superhelix. It should also be possible to determine the mass per unit length and hence the superhelical pitch from the unit cell dimensions of the liquid crystalline phase. Supercoiled, nicked and linear forms of a number of plasmids are being studied and compared.

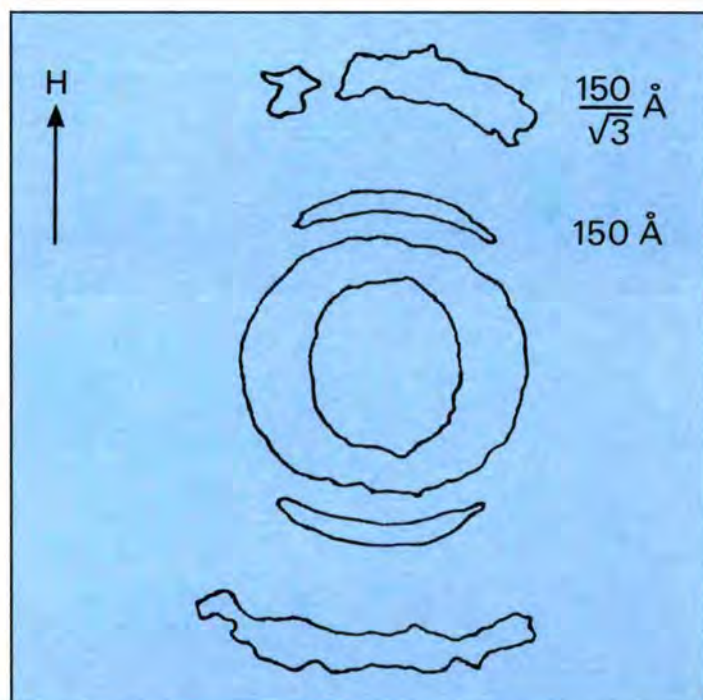


Figure 67: Contour plot of the low-angle diffraction obtained on D11 from a liquid-crystalline solution of superhelical plasmid ( $\sim 30\text{mg/ml}$ ) in D<sub>2</sub>O buffer. The sample was oriented in a magnetic field  $H$ , (direction shown) of 5 Tesla prior to measurement. The pattern shows the (1,0) and (1,1) reflections of a two-dimensional hexagonal lattice.

The jointly EMBL/ILL funded low resolution diffractometer DB21 has been used to collect data on several different single crystal complexes. Data were collected to 16 Å from crystals of the swollen form of tomato bushy stunt (TBSV) virus. These should give information on how the interactions between the RNA and internal protein change on swelling. This will complement the recent studies on native TBSV which identified some of the specific sites of RNA/protein interaction. [Fig. 68]

In a single crystal study of the lipoprotein lipovitellin (St. Louis, ILL, EMBL) DB21 was used to collect data to 12 Å resolution, the highest resolution to which data have so far been collected on this instrument. Lipovitellin is a serum lipoprotein of molecular weight ~ 350 000 containing some 15% phospholipid. The object of the DB21 study is to locate the phospholipid which in high resolution X-ray maps is invisible due to disorder and low contrast.

Further measurements have been carried out on single crystals of the 50S ribosomal sub-unit (Hambourg, MPI Berlin, LURE, EMBL, ILL). Recent improvements in crystal quality have allowed good quality data to be measured to about 25 Å resolution.



Figure 68: RNA packing in tomato bushy stunt virus. The red-contoured density shows a piece of RNA interacting with basic amino acids on the inside of the protein shell of the virus. Data obtained on D17 and picture produced on the Evans and Sutherland PS300 display system.

Secretary: P. Timmins  
Head of EMBL Outstation: B. Jacrot

# COLLEGE 9 CHEMISTRY

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P. Lindner  
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P. Terech  
A. Wright

## INTRODUCTION

The scientific work in the college was again very productive and the two short-period shut-downs proved useful to finish off work. The systems investigated and experiments carried out continue to become more elaborate, increasing the demand for non-standard sample environments. With the enhancement of the central computer foreseen for 1988, we hope to improve the data-treatment.

## SCIENTIFIC HIGHLIGHTS IN 1987

### Molecular Spectroscopy, Surfaces and Mesophases

#### Quantum and rotational motions

The first examples of metal complexes which bind hydrogen in MOLECULAR (dihydrogen) rather than dissociated (dihydride) form have been investigated by means of rotational tunnelling spectroscopy of the hydrogen molecule. These systems provide an opportunity to study the simplest chemical bond, the H-H bond, in an arrested intermediate state of the oxidative addition of H<sub>2</sub> to a metal center. It is therefore of considerable interest to determine to what extent this unique three-center metal dihydrogen bond can be probed by studies of the reorientations of the bound hydrogen molecule. Initial experiments on W(CO)<sub>3</sub>H<sub>2</sub>(PCy<sub>3</sub>)<sub>2</sub> (where Cy = C<sub>6</sub>D<sub>11</sub>) using IN6 provided convincing evidence for rotational tunnelling of

H<sub>2</sub> (Los Alamos, ILL). The measured tunnel splitting of 0.120 meV could be analysed in conjunction with INS torsional data obtained at the Los Alamos Neutron Scattering Center in terms of planar rotation normal to an axis through the W atom and the midpoint of the H-H bond. Both crystallographic and theoretical studies find only one equilibrium orientation for the H<sub>2</sub> molecule, so that the rotational potential can be assumed to be of the form:

$$V = \frac{1}{2} V_2 \cos 2\Phi.$$

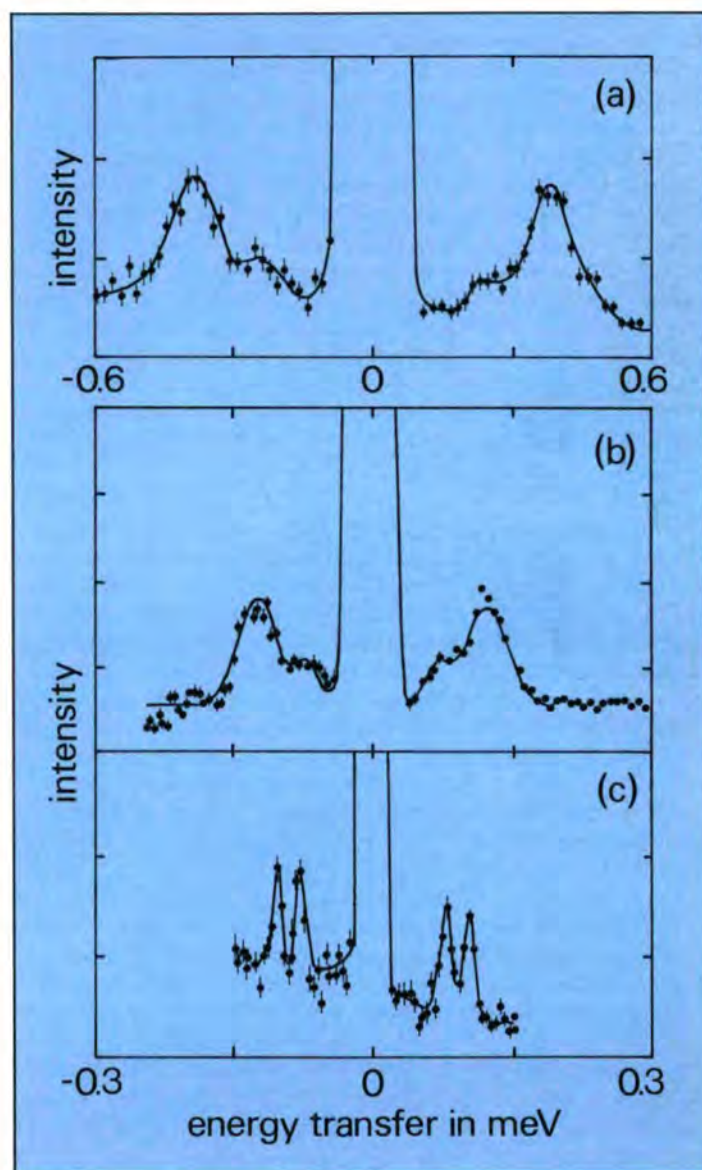


Figure 69: Tunnel splitting of  
(a) Mo(CO)<sub>3</sub>H<sub>2</sub>(PCy<sub>3</sub>)<sub>2</sub>, λ = 6 Å  
(b) W(CO)<sub>3</sub>H<sub>2</sub>(PCy<sub>3</sub>)<sub>2</sub>, λ = 8 Å  
(c) W(CO)<sub>3</sub>H<sub>2</sub>(P-iPr)<sub>2</sub>, λ = 10 Å

The barrier height derived from this analysis is 15.4B or 94.5 meV, where the stretched H-H bond length has been taken into account. High resolution data taken subsequently on IN5 and IN13 on the same system (Fig. 69b) revealed a split tunnelling peak, which may be attributed to disorder known to be present in the PCy ligands. In view of this observation one could assume that the rotational potential for H<sub>2</sub> is largely determined by the bulky ligands of the metal atom. The opposite,

however, turns out to be the case. Data taken with the Mo analog of the above, i.e.  $\text{Mo}(\text{CO})_3\text{H}_2$  ( $\text{PCy}_3$ )<sub>2</sub>, shown in Fig. 69a, reveal the same spectrum as the W- compound except at a frequency nearly three times as high. This implies a lower barrier for  $\text{H}_2$  rotation which is also in accord with the known weaker metal-dihydrogen binding and lesser thermal stability of the Mo compound. Moreover, data taken with another W-compound with different ligands,  $\text{W}(\text{CO})_3\text{H}_2$  ( $\text{P-iPr}_3$ )<sub>2</sub> where  $\text{i-Pr} = \text{C}_3\text{D}_7$ , show a different spectrum (Fig. 69c) (again with two peaks, but intensity ratio 1:1 rather than 1:3) but the MEAN transition frequency is only somewhat less (0.09 meV vs. 0.11 meV) than that for the W-compound with  $\text{PCy}$  ligands. These data clearly suggest that the observed tunnel splittings for  $\text{H}_2$  in these systems is PRIMARILY a measure of the metal-dihydrogen interaction, and that the fine structure of the tunnelling spectrum is associated with the bulky ligands. The function of the latter may be to prevent the oxidative addition reaction from going to completion. The above conclusions are also in accord with the theoretical picture of the electronic bonding of metal-dihydrogen complexes. In these calculations the orientation of the  $\text{H}_2$  molecule relative to the metal complex is determined by the optimisation of the electron transfer between the metal and  $\text{H}_2$  that stabilise the dihydrogen binding. These systems which are of enormous significance in Chemistry represent therefore a relative unique case of where the well known sensitivity of rotational tunnelling spectroscopy can be utilised as a rather direct probe of chemical bonding.

The low-temperature rotational motions of  $\text{NH}_3$ -groups in a variety of transition-metal hexammine ions have now been characterised in some detail (ILL). In the extreme case, transitions between "free-rotor" energy-levels are observed in the case of  $\text{Co}(\text{NH}_3)_6(\text{PF}_6)_2$  demonstrating that the inherent intramolecular interactions are extremely small. As the size of the anion is decreased, motions of the  $\text{NH}_3$  groups become more hindered and are then best described by rotational tunnelling.  $\text{Ni}(\text{NH}_3)_6\text{I}_2$  provides a convenient example for study since the tunnelling spectrum falls in the range of energy-transfers most easily accessed by neutron scattering. The spectrum is rather complex, and despite experimental and theoretical effort, the details of its origin are not yet completely understood. Nevertheless, the spectral changes resulting from changes in pressure, temperature and partial isotopic substitution show that the rotational hindrance potential is dominated by neighbouring hexammine clusters and that  $\text{NH}_3$ -reorientation on a given cluster is a cooperative event.

The higher rotational barrier of the nitrate analogues provides a system in which the rotational diffusion of the  $\text{NH}_3$  groups can be measured using QNS. The overall radius of rotation is appreciably greater than the 0.9 Å expected for uniaxial  $\text{NH}_3$  rotation and it seems certain that some motion of the whole cation is also involved, possibly to minimise the interionic  $\text{NH}_3:\text{NH}_3$  interactions alluded to above. The picture of molecular motions within the various phases of this compound are summarized in Figure 70 which shows the variation of the fraction of protons in the system contributing to the EISF with temperature. QNS sees the motions of the  $\text{NH}_3$  groups on one of two cation sublattices in phase III. As the sample-temperature is increased, one sublattice becomes increasingly disordered as the III-II transition is approached. Phase II is marked by the onset of  $\text{NH}_3$  disorder of the second cation sublattice seen by the appearance of a second quasi-elastic component. Complete disorder of the second

sublattice coincides with the II-I transformation in which a cubic lattice ( $\text{Fm}\bar{3}\text{m}$ ) is formed.

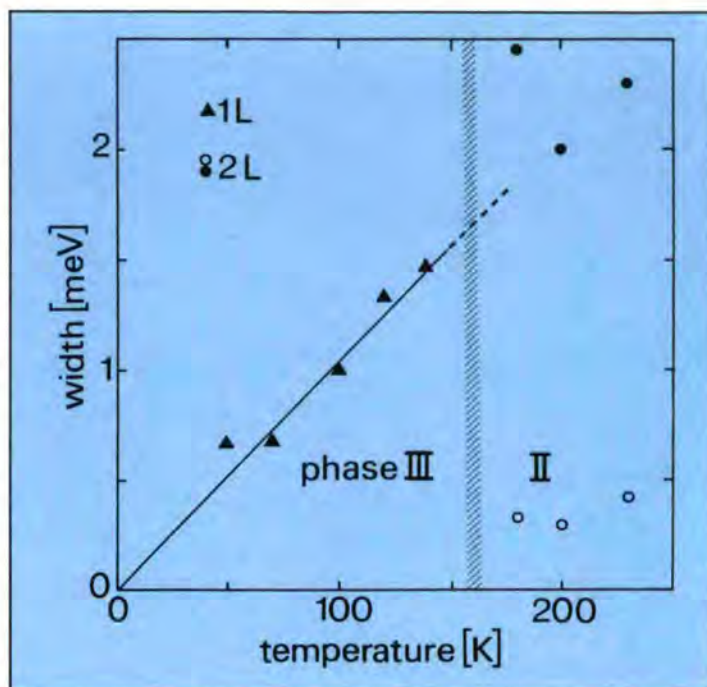


Figure 70: Variation of peak-width (diffusion constant) derived from the quasielastic components.

### Quasi-elastic scattering

The rotational dynamics of methanol and benzene adsorbed in ZSM-zeolite have been studied by quasi-elastic neutron scattering (QNS) (Villeurbanne, ILL). Two molecular species are considered in each case, in order to account for the variation of the EISF. There are almost fixed molecules, whether adsorbed by hydrogen bonds in the case of methanol or blocked by steric hindrance of benzene. There are also mobile molecules, differing within a sphere of diameter  $\sim 4.8$  Å in the case of methanol, or performing uniaxial rotational diffusion for benzene.

The family of  $\text{NH}_4\text{MF}_3$  perovskites ( $\text{M} = \text{Mg}, \text{Zn}, \text{Mn}, \text{Co}, \text{Cd}$ ) has been extensively studied with calorimetry-techniques, NMR and Raman and IR spectroscopy. Heat capacity measurements show a large anomaly due to the reorientations of the  $\text{NH}_4^+$  ions. This first order phase transition takes place at critical temperatures which decrease with decreasing cell dimensions ( $\text{Cd} \rightarrow \text{Mg}$ ). Upon cooling, the symmetry is reduced from  $\text{Pm}\bar{3}\text{m}$  to pseudo-tetragonal with  $c > a$ . An electrostatic point-charge model has been proposed to explain the behaviour of the  $\text{NH}_4^+$  ion. It predicts decreasing rotational hindering barriers with increasing cell dimensions. However, recent NMR experiments have shown the opposite trend in the activation energies, suggesting repulsive forces and hydrogen bonding F-H. QNS spectra have been collected for some of the compounds in order to determine the activation energies and jump geometry (Zaragoza, ILL). Increasing activation energies with increasing cell dimensions have been obtained assuming an exponential dependence of the quasi-elastic linewidth broadening with the inverse temperature, showing the same trend as in the NMR experiments. Besides, a step in the quasi-elastic widths suggests jumps which do not leave the  $\text{NH}_4^+$  group invariant. A simple  $90^\circ$  jumps model is

suitable for temperatures above  $T_c$ , but fails for the low temperature phase, and a more complicated model has to be used.

The step in the quasi-elastic widths turns out to be approximately the same ( $\sim 70 \mu\text{eV}$ ) for  $\text{NH}_4\text{MnF}_3$  and  $\text{NH}_4\text{ZnF}_3$  (Figure 71). Therefore, this step seems to be independent of the M ion substitution and should be related to slow varying parameters of the interaction of an  $\text{NH}_4^+$  group with its surroundings.

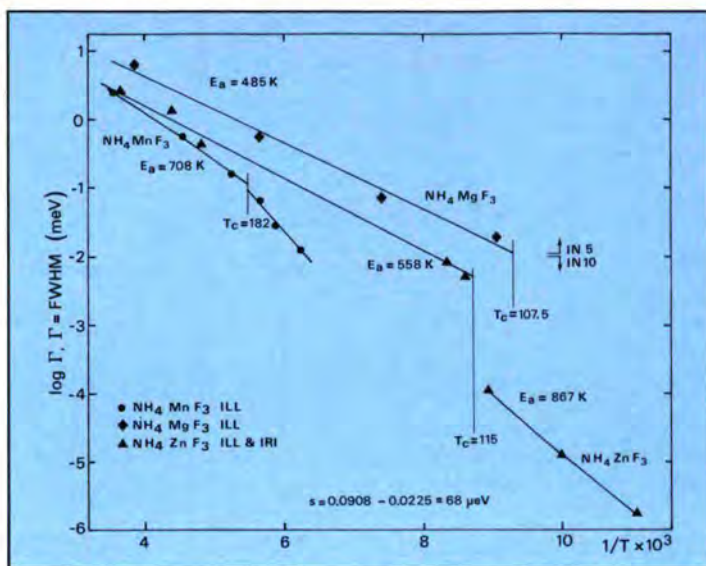


Figure 71: Logarithm of the quasielastic linewidth broadening versus  $1/T$ . Data:  $\text{NH}_4\text{MnF}_3$  and  $\text{NH}_4\text{MgF}_3$ : INS (ILL);  $\text{NH}_4\text{ZnF}_3$  and  $T < T_c$ : IN10 (ILL);  $\text{NH}_4\text{ZnF}_3$  and  $T > T_c$ : RSK1 (IRI).

The  $\text{NH}_4\text{MnCl}_3$  perovskite has the same crystallographic structure as the fluorine family, but shows only a small jump in the heat capacity measurements. Here the substitution is the halide, and at present it is being investigated whether there is also a step in its relaxation times. It has been proposed as an example for  $\text{NH}_4^+$  free rotor, and such a step should not appear.

The aim of the present study is to investigate the dynamics of alkyl-chains  $-(\text{CH}_2)_n-$  connected to the solid-solid phase transition phenomena in molecular crystals, such as the perovskite-type layered compounds alkylene-diammonium  $[\text{NH}_3-(\text{CH}_2)_n-\text{NH}_3]\text{MnCl}_4$  ( $2\text{C}_n\text{Mn}$ ) and alkyl-ammonium  $[\text{NH}_3-(\text{CH}_2)_n-\text{NH}_3]\text{MnCl}_4$  ( $\text{C}_{n+1}\text{Mn}$ ), the  $n$ -alkane  $\text{CH}_3-(\text{CH}_2)_n-\text{CH}_3$  compounds (Bordeaux, ILL). For the alkylene-diammonium (in short  $2\text{C}_n\text{Mn}$ ), the spectra were recorded using the three  $\text{NH}_3(\text{CH}_2)_n\text{NH}_3$ ,  $\text{ND}_3(\text{CH}_2)_n\text{ND}_3$  and  $\text{NH}_3(\text{CD}_2)_n\text{NH}_3$  derivatives. In such a structure, the two ends of the cations are bounded via hydrogen bonds to the inorganic matrix and the chain keeps an "all trans" rigid structure only for  $n=3$  while jumps over two "twisted" and two "trans" states must be postulated to take into account both large and small amplitude jumps of the protons revealed by the quasi-elastic spectra of the  $2\text{C}_4\text{Mn}$  and  $2\text{C}_5\text{Mn}$  compounds. The  $-\text{NH}_3^+$  polar heads show, in addition with the  $90^\circ$  jumps connected to the motions of the  $-(\text{CH}_2)_2-$  skeleton, independent oscillations around the  $\text{C}-\text{NH}_3$  axis. The "lifetime" as well as the population of "twisted" conformers increases as the temperature raises. In the alkyl-ammonium systems  $[\text{NH}_3-(\text{CH}_2)_n-\text{CH}_3]\text{MnCl}_4$  ( $\text{C}_n\text{Mn}$ ), the chains are bounded to the host lattice by one end only and can be very well compared to lipidic membranes. The QNS experiments

were performed on the fully hydrogenated  $\text{C}_{10}\text{Mn}$  and the partially deuterated  $[\text{ND}_3-(\text{CD}_2)_n-(\text{CH}_2)_8-\text{CH}_3]\text{MnCl}_4$  ( $\text{C}_{10}\text{Mn}-\text{cd}_2\text{-nd}^3$ ) compounds. At the solid-solid phase transition, a very strong change in the quasi-elastic spectrum is evidenced; the amplitude of the motions is very large and results from fast transitions between various conformers involving gauche and kink defects. Here, the dynamical process cannot be longer interpreted in terms of instantaneous jump process. In the case of  $n$ -nonadecane ( $n=17$ ), we investigated by means of QNS the rotator  $\text{R}_I$  phase of the fully hydrogenated and its deuterated  $\text{CD}_3-(\text{CH}_2)_{17}-\text{CD}_3$  and  $\text{CD}_3-\text{CD}_2-(\text{CH}_2)_{15}-\text{CD}_2-\text{CD}_3$  compounds. The use of semi-oriented samples allowed us to discriminate between motions occurring along the molecular axis and perpendicular to it. It is seen that fast restricted translational motions occur in the chain direction in connection with a twisting of the ethyl-end groups while the "body" performs rotational fluctuations around the main molecular axis. For longer molecules ( $n=23, 24$ ), the amplitude of the motions increases and the uniform rotation limit seems to be nearly reached in the high temperature  $\text{R}_{II}$  phases.

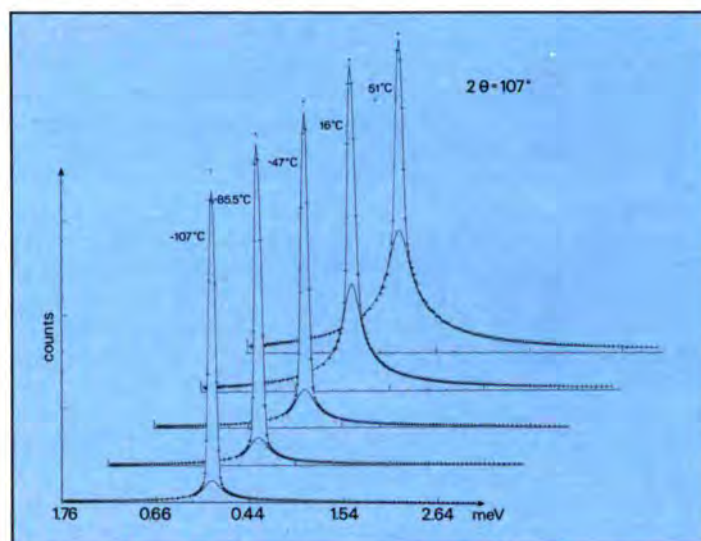


Figure 72: Quasi-elastic spectra of  $\text{C}_{18}\text{O}_2/\text{Ud}_4$  at various temperatures

A further part of the general study of the dynamics of alkyl-chains deals with the molecular motions of these chains trapped within solid urea inclusion compounds (Bordeaux, ILL). In urea inclusion adducts of long chain compounds, the urea host crystallises in an orthorhombic structure at low temperature and there is a solid-solid phase transition to a hexagonal system as the temperature raises. The guest molecules are ordered in the low temperature phase while a dynamical order-disorder transition occurs at the phase transition. The interesting point in such a system is that the molecules of urea form infinitely long channels in which the alkyl-chains are forced (due to the small channel diameter) to take up "all-trans" conformations. The rotational and translational motions can then be investigated without the effects of conformational dynamics. No quasi-elastic broadenings can be evidenced within the experimental resolution ( $70 \mu\text{eV}$ ) in the low temperature orthorhombic crystalline phase. The phase transition to the hexagonal structure is expected between  $-114^\circ$  and  $-104^\circ \text{C}$ . From  $-117^\circ$  up to  $51^\circ \text{C}$ , there is evidence for large quasi-elastic broadenings (Fig. 72). An experimental Elastic Incoherent Structure Factor (EISF) can be evaluated

showing that the amplitude of the motions increases continuously as the temperature raises. In a first approximation, the dynamics of the chain cannot be pictured as a single jump process between several equivalent sites, but originate more likely in some kind of oscillatory behaviour about the main molecular axis. In addition to this process, translational motions are expected to occur in the chain direction. The spectra recorded in the dynamically disordered phase of this compound are very similar to those of adducts of n-nonadecane, and the resulting EISF can be interpreted in a similar way. The dynamical behaviour of the adducts of n-nonadecane and stearic acid look similar, the main interesting feature observed is that the amplitudes of the motions are temperature dependent. A first estimate of the quasi-elastic broadenings by two lorentzians show that two kinds of motions can be detected, a slow one occurring on a 10 ps time scale and a fast one on a 4ps time scale (values obtained for both compounds at the highest temperatures). It is however not yet clear if these lorentzians originate from two uncorrelated motions (rotation/translation) or from a diffusive-type rotational process.

An investigation of the disordered phases of decylammonium chloride  $C_{10}H_{21}NH_3Cl$  gives information about the slow motions of the alkyl chains in layered organic crystals (Bordeaux, ILL). This compound crystallises in a bidimensional structure constituted of alternative sheets of chlorine matrix and of aligned organic chains. The ammonium ends of adjacent chains are linked through hydrogen bonds to the chlorine atoms. In the low-temperature ordered phase  $\epsilon$ , the chains are intercalated. As the temperature increases, this compound undergoes thermotropic phase transitions involving a desintercalation of the chains and leading to highly disordered phases. The desintercalation process is "irreversible" (the  $\epsilon$  phase transforms to  $\delta$  phase after several days). From the runs obtained with the IN13 spectrometer, the Fig. 73 corresponding to the instrument time-scale can be calculated by means of one lorentzian function and the quasi-elastic broadenings can also be evaluated. The following results are obtained:

- in phase  $\epsilon$ , there is evidence for no quasi-elastic broadening,
- the  $\delta$  phase, however, displays a nice broadening corresponding to a characteristic time of about 30 ps,
- in the  $\alpha$  phase, motions on a time-scale of about 20 ps are detected, their amplitude is increasing drastically as compared to phase  $\delta$ . In conjunction or related to these motions, another process occurring on a 170 ps time-scale can be evidenced on the IN10 spectrometer.
- in the  $\epsilon$  phase, we do observe QE broadenings which can be compared to those observed in phase  $\delta$ .

Two main conclusions can be drawn from this first evaluation of the data; the motions are all occurring in conjunction with a conformational disorder and can be related to that; the fact that no broadening can be observed in the  $\epsilon$  phase confirm this assumption. The amplitude of the motions increases as the conformational disorder raises while the characteristic times seem to be temperature dependent. It is likely that fast processes also related to the conformational disorder occur in these phases on a picosecond time-scale (as observed in urea inclusion compounds).

The dynamics of alkyl chains in Cu alkanolate complexes in their discotic phases have also been investigated (Lille, CENG, ILL): i.e.  $Cu_2(C_{16}H_{31}O_2)_4$ ,  $Cu_2(C_{15}H_{29}O_2)_4$ ,  $Cu_2(C_{12}H_{23}O_2)_4$ ,  $Cu_2(C_9H_{17}O_2)_4$ ,  $Cu_2(C_6H_{11}O_2)_4$ . Three main conclusions could be drawn from these experiments.

a) Clearly there is an evidence of large amplitude motions not only in the high temperature discotic phases, but also in the crystalline phases.

b) The mean amplitude of the motion continuously increases with increasing length of the chains.

c) The characteristic time of the motions, and also their amplitude, are temperature-dependent in the discotic, super-cooled discotic, and crystalline phases.

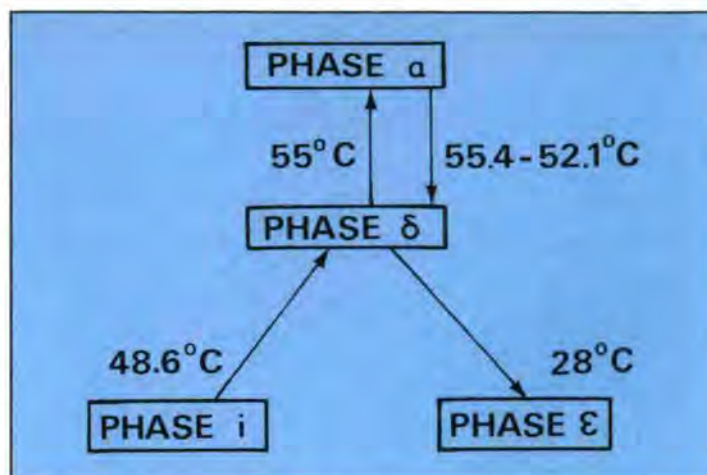


Figure 73: Phase transitions sequence in  $C_{10}H_{21}NH_3Cl$

$CsHSO_4$  is a particular case of extensive study that was performed on a large family of solid state protonic conductors of general formula  $MHXO_4$  where  $X = S$  or  $Se$  and  $M =$  alkali metals,  $NH_4$ ,  $N(CH_3)_4$  and even amines such as protonated pyridine or biprotonated triethylene-diamine or polyethylene imine (Bordeaux, Palaiseau, Grenoble, ILL). For the two latter compounds,  $Py$ ,  $H_2SO_4$  it can be shown that the cation undergoes reorientational motions, even at room temperature, which certainly helps the migration of the  $HSO_4^-$  proton. However, the large number of protons held by these molecules prevents a precise analysis of the  $HSO_4^-$  dynamics itself to be performed.  $CsHSO_4$  seems to be an ideal case for the study of the conduction mechanism in an anhydrous solid conductor since the proton of interest dominates by far the scattering of the other atoms. A first experiment on IN10 indeed shows that the superionic phase presents a liquid-like behaviour and allows values of the proton self-diffusion constant of  $1.1 \cdot 10^{-7}$  to  $1.8 \cdot 10^{-7} \text{ cm}^2\text{s}^{-1}$  to be deduced respectively between 420 to 450 K, in very good agreement with the NMR determination. To our knowledge it is the first time that a high resolution neutron scattering experiment succeeds in characterizing clearly the proton diffusion in an anhydrous solid conductor.

QNS experiments were performed to understand the molecular mechanism of the enhanced gas solubilisation in liquid fluorocarbons (Nancy, ILL). A special pressure cell was built (thickness 5 mm) allowing a preliminary study of the perfluorinated heptane saturated with  $CH_4$  at pressures till 30 bars and for temperatures in the range  $-40^\circ\text{C}$  to  $20^\circ\text{C}$ . The diffusion of the gas appears to be noticeably slower in the fluorinated liquid than in the hydrogenated one.  $D_T$  depends very little on the pressure, but markedly changes with the temperature: from two experimental values,  $D_T$  seems to be proportional to  $(T - 200 \text{ K})$ , suggesting a diffusion equal to zero at the melting of  $C_7F_{16}$  ( $\sim 200 \text{ K}$ ).

## Vibrational spectroscopy

The inelastic neutron scattering spectra (INS) of hydrogen adsorbed on small particles of Palladium encaged in a Y-Zeolite has been studied as a function of  $H_2$  pressure (Villeurbanne, ILL). It has been shown that the hydrogen atom begins to occupy subsurface sites before the saturation of the chemisorption sites. The formation of the hydride phases is progressive and they have the same vibrational frequencies as in the bulk.

The INS spectra of solid and adsorbed benzene have been compared (Villeurbanne, ILL). The INS intensities of solid benzene have been derived from a force field calculation, including multiphonon processes. After adsorption in Na-Y, two types of adsorbed benzene molecules have been found, which is in agreement with diffraction work. Small frequency shifts indicating a weak perturbation of the benzene ring have been observed.

## Reflection from surfaces

For many years neutrons have been used to study the structure and dynamics of bulk samples, the lack of charge has allowed greater penetration than alternative probes such as photons or electrons. In recent years a particular application to the study of surfaces has developed - this is the measurement of reflectivity profiles which gives specific information about the distribution of scattering length density perpendicular to the surface. Similar experiments can, of course, be performed with X-rays but the special contrast available to neutrons (isotopic labelling, spin analysis) have made this technique popular.

plicated experiments, excess polymer at the solution - air interface (Oxford, Salford, Strathclyde, ILL). Some results of this last experiment are shown in Figure 74. From this data it is concluded that there is an excess of polymer at the interface in a diffuse layer about  $80 \text{ \AA}$  thick. These measurements were made on D17 which has a large two dimensional multidetector which permitted simultaneous measurements of the diffuse scattering around the reflected beam.

Another experiment on PVC lattices followed the structural changes in film formation during  $\langle \rangle$  with scattering measurements in reflection geometry (Salford). This provided a useful complement to scattering measurements in the usual transmission geometry.

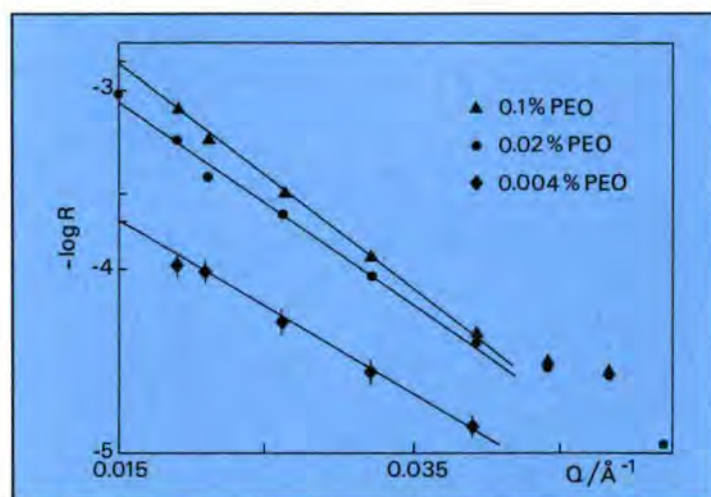


Figure 74: Reflection profile of polyethylene oxide (PEO) dissolved in contrast matched  $H_2O/D_2O$  at three different concentrations. Notice the large dynamic range covered in these measurements.

Some measurements from the ILL were published 10 years ago; other research centres have now developed new instruments particularly for this type of work. However, there is still a significant amount of work in this field at the ILL, particularly exploiting the high flux available at long wavelengths. One area of interest is the study of this magnetic films (this falls outside the domain of College 9). The largest interest has been in the fields of chemistry and materials science; several groups have been active.

Samples have included  $\langle \rangle$ -Blodgett films (Bristol), hard carbon surfaces (Bristol, Wilton) and is perhaps the most com-

## Phonons in the commensurate phase of D<sub>2</sub> on graphite

An adsorbed monolayer of atoms or molecules on a substrate is highly interesting because it simulates a two dimensional (2-d) system. In some cases the adsorbate can be considered as a free floating layer on the substrate. In other cases a strong interaction between adsorbate and substrate occurs. Both situations are found in the phase diagram of deuterium (D<sub>2</sub>) adsorbed on graphite<sup>1)</sup>: almost free floating behaviour is observed near the densest monolayer whereas locking is seen in the  $(\sqrt{3} \times \sqrt{3})R 30^\circ$  commensurate phase around  $2/3^{\text{rd}}$  of the density of the densest monolayer. Figure 91 shows a top view of this commensurate structure. The repulsive contribution of the zero point motion of D<sub>2</sub> makes this registry attainable. A study of the phonons in the different phases gives information about the changing interactions between adsorbate and substrate. Here we want to concentrate only on the commensurate phase to investigate the ratio between D<sub>2</sub> - D<sub>2</sub> interaction and D<sub>2</sub> - substrate (graphite) interaction.

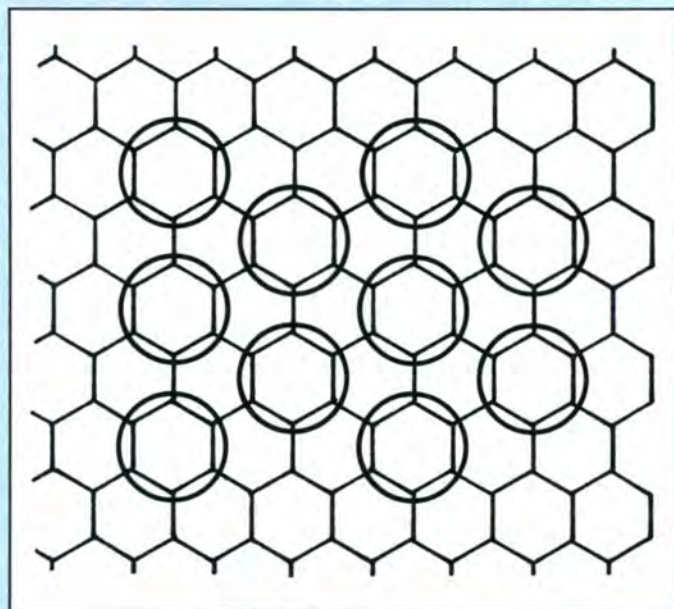


Figure 91: Model of the  $(\sqrt{3} \times \sqrt{3}) R 30^\circ$  commensurate phase of D<sub>2</sub> on graphite

It should be noted that the commensurate-incommensurate transition between these two phases gives rise to some intermediate phases which exhibit domainwalls (striped superheavy - hexagonal heavy - or highly mobile ones<sup>2)</sup>).

Neutron scattering is well suited to look for dynamical properties in solid state physics. Interfaces and surfaces can also be investigated. However, the application to (2-d) systems like adsorbates on substrates is difficult because a large surface area is needed to obtain a large signal. In the case of graphite as a substrate this difficulty is overcome by using exfoliated graphite. This substrate can offer about 200 m<sup>2</sup> of surface area in a neutron scattering sample. The price for this large surface area is the random orientation within the (001) plane and the distribution of about 30° (FWHM) of the (001) direction. Thus a scan in diffraction or in inelastic scattering exhibits always an orientational average due to these substrate properties.

Two inelastic scans are shown in figure 92. A difference contrast is displayed: the counts from the sample cell without the adsorbate have been subtracted from the counts with the adsorbed D<sub>2</sub>. The scans have been taken on IN3 (elastic resolution 0.033 THz FWHM) at two different momentum transfers. A clear signal given from the D<sub>2</sub> molecules on the graphite is seen. A shift of the signal from around 0.9 THz to slightly more than 1.0 THz is observed if the Q-transfer is reduced from 1.7 Å<sup>-1</sup> to 0.85 Å<sup>-1</sup>, respectively. Thus a first conclusion is that no complete "Einstein" - behaviour is present, which would give a Q-independent energy transfer. However the average energy transfer of about 1 THz is very close to 1.036 THz calculated for a pure D<sub>2</sub>-graphite interaction<sup>3)</sup>.

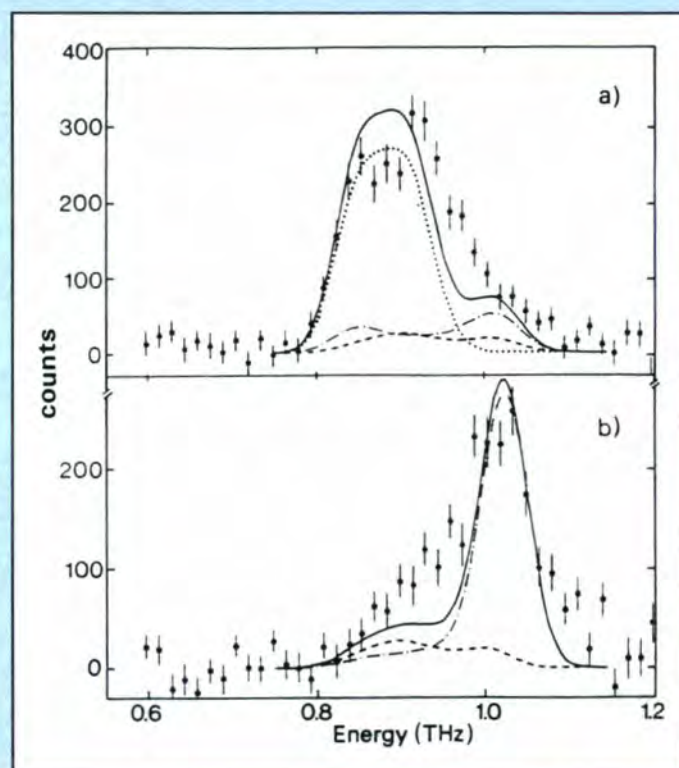


Figure 92: Inelastic neutron scattering data for  $Q = 1.70 \text{ \AA}^{-1}$  (a) and  $Q = 0.85 \text{ \AA}^{-1}$  (b) for D<sub>2</sub> on graphite in the commensurate phase (the background has been already subtracted). The full line shows the calculated lineshape, the dotted line is the contribution from the transverse mode, the dashed-dotted line is the contribution from the longitudinal mode and the dashed line is the incoherent scattering.

The Q-dependence of the energy transfer in figure 92 must be due to the interaction between the D<sub>2</sub> molecules even being pinned to registry. In a simple model the D<sub>2</sub> molecules are connected by springs (only to next neighbours) in a triangular network and by a spring to the graphite. With this model the dispersion curves of the D<sub>2</sub> phonons are calculated and they are displayed in figure 93. The two spring constants are 0.0080 N/m (to next neighbour) and 0.091 N/m (D<sub>2</sub> to substrate)<sup>3)</sup>. Thus the interaction between the D<sub>2</sub> molecules is really weak in comparison to the one between D<sub>2</sub> and the substrate. It should be noted that the scale in figure 93 is very much expanded and that the zero is not shown to scale. The dispersion curve is indeed very flat.

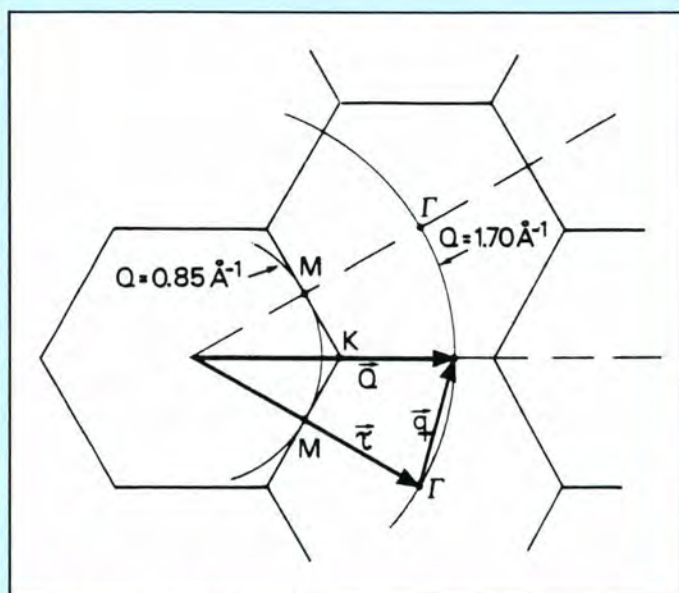


Figure 93: The region of reciprocal space covered by the scans which are shown in fig. 92. A scattering-triangle is drawn as an example. It represents a case of excitation of a transverse zone boundary phonon.  $Q$  is the total momentum transfer;  $\tau$  is a reciprocal lattice vector and  $q$  is the phonon momentum.

In order to understand how the dispersion curves and the spring constants have been calculated we describe now the scans taken at different  $Q$  in reciprocal space. This is explained in figure 94. The distance to the first  $\Gamma$  points is  $1.7 \text{ \AA}^{-1}$  just the value chosen for the first scan in figure 92. The measured signal consists of a directional average along the circle with radius of  $1.7 \text{ \AA}^{-1}$  (taking into account the instrumental resolution) from all phonons for which the scattering triangle closes. Those phonons for which a singularity in the density of states exists along this path contribute most to the signal. Thus this scan focusses on the phonons at the zone centre and the transverse phonons at the zone boundary. The scan taken at  $Q = 0.85 \text{ \AA}^{-1}$  for which the path in reciprocal space is also shown in figure 94 focusses on the longitudinal zone boundary phonon. This explains the shift of the signal using  $1.7 \text{ \AA}^{-1}$  or  $0.85 \text{ \AA}^{-1}$  in momentum transfer: because the density of states of the longitudinal phonons culminates at a higher value compared to the one of the transverse phonons. The full lines in figure 92 display the calculated lineshape of the spectrum at the two momentum transfers  $Q$  for the irreducible  $1/12$ th of the Brillouin zone (figure 94) with the dispersion relation (figure 93) taking into account the 2-d averaging, the mosaic spread around the (001) direction of the graphite and the resolution ellipsoid of the spectrometer. Finally the two spring constants (which give the dispersion curves in figure 3) have been varied until a best fit was obtained for the leading edge of the excitation at the phonon-gap (at  $0.85 \text{ THz}$  in fig. 92a) and for the longitudinal zone boundary phonon (main peak at  $1.01 \text{ THz}$  in fig. 92b). The different line types in figure 92 give the contributions of the transverse and longitudinal phonons and the one of the incoherent scattering.

The present data show unambiguously that dispersion is present in the system of  $D_2$  in the commensurate phase on graphite. The coupling to neighbouring molecules is nearly ten times smaller than the coupling to the substrate (in the in-plane direction !). For the first time the phonon gap was

shown to exist for a system of a simple physisorbed molecule. The separation of the excitations at the phonon gap from those at the zone boundary (transverse phonons in figure 92a) is still more pronounced in the data than in the calculation. This may be improved by considering the coupling to further neighbours. However, a clear distinction was made between the phonons shown in figure 92a with respect to those shown in figure 92b due to the correct choice of the momentum transfer. In figure 92a the intensity from the phonons around the phonon gap and the intensity from the transverse zone boundary phonons are shown whereas in figure 92b the intensity from the longitudinal zone boundary phonons is separately displayed.

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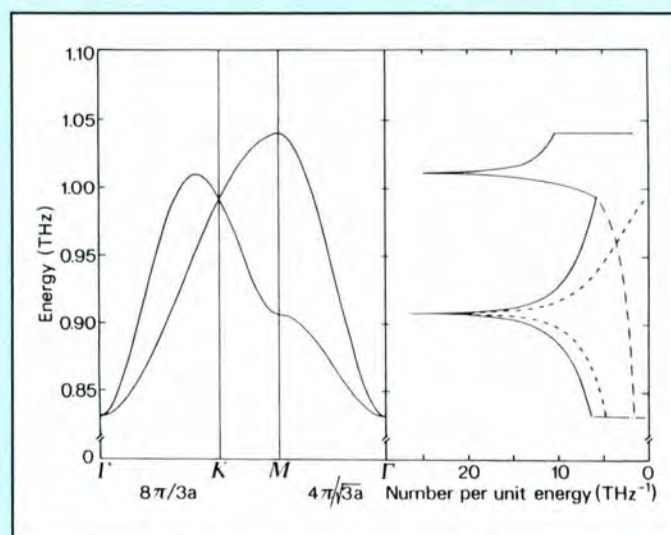


Figure 94: Dispersion relation for the commensurate triangular lattice of  $D_2$  on graphite (on the left) and the corresponding density of states (on the right). The different branches are the longitudinal and transverse in plane modes. The out of plane mode has a much higher energy and is an Einstein mode in this model (notice the expanded scale).

- [1] Freimuth and H. Wiechert, *Surf. Sci.* 178, 716 (1986)
- [2] H. P. Schildberg, H. J. Lauter, H. Freimuth, H. Wiechert, and R. Haensel, *Proceed. LT-18, Jap. J. Appl. Phys.* 26, Suppl. 26-3, 345 (1987)
- [3] V. L. P. Frank, H. J. Lauter, and P. Leiderer, *Proceed. LT-18, Jap. J. Appl. Phys.* 26, Suppl. 26-3, 347 (1987) and to be published.

## Colloids and polymers

Some selected topics of the numerous neutron scattering experiments on colloids and polymers are briefly reviewed below. As usual, most of them were performed at the small angle scattering instruments D11 and D17, the remaining part of work in this field was carried out at the quasi-elastic spectrometers, such as the spin-echo machine IN11, and other diffractometers.

### Inelastic neutron scattering

After the success of the Neutron Spin Echo method concerning the investigation of the dynamics of simple linear polymers (dilute, semi-dilute solutions, melts) the interest has turned towards the more complicated systems like star-polymers (ILL, Exxon, Mainz), networks (Strasbourg, ILL), block copolymers (ILL, Freiburg, Strasbourg), where the specific labelling and contrast variation allows the study of the overall and the relative dynamics of the different parts of the molecules. The microemulsion systems stay in the focus of interest as looking for glass transition in dense systems or as investigations of the relevant parameters in droplet shape-fluctuations (one of the most interesting findings of the last year) in different systems (ILL, Exxon).

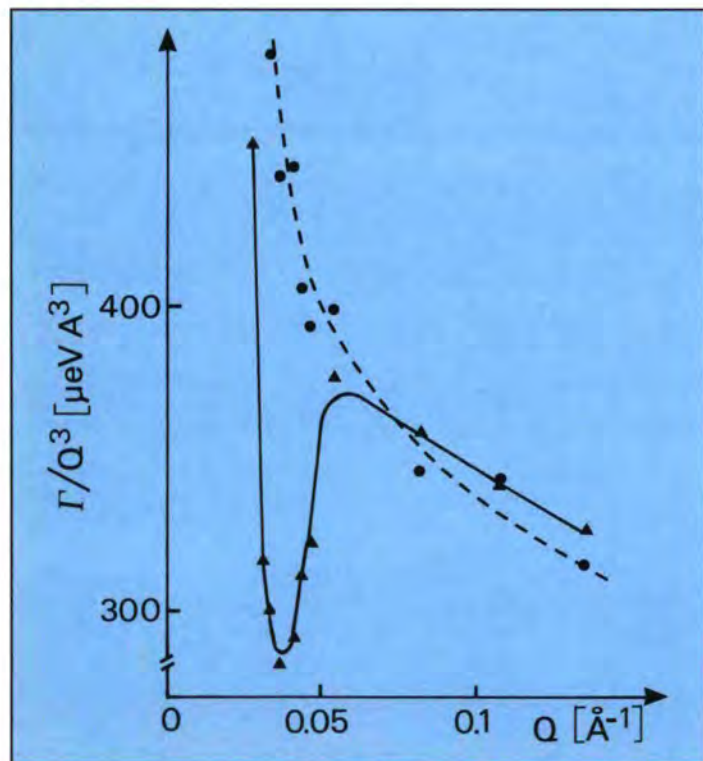


Figure 75: Reduced relaxation rates  $\Gamma/Q^3$  vs  $Q$  from a fully labelled 12-arm PS-star ( $\blacktriangle$ ) and deuterated 12-arm PS-star with one labelled arm ( $\bullet$ ). The dashed and solid lines are guides for the eye.

### Collective vs single arm relaxation of star polymers

Recent neutron spin echo experiments on polyisoprene stars in dilute solution revealed a strong interrelation between dynamic response and star architecture. It manifests itself in a deep minimum in the reduced Zimm relaxation rate  $\Gamma Q^3$  around  $QR_g^{\text{arm}} \approx 1.5$  where  $R_g^{\text{arm}}$  is the radius of gyration of one arm. The occurrence of this minimum was interpreted as resulting from a De Gennes type narrowing in the collective internal dynamics of the star and was related to the strong

enhancement of the radial segment distribution function in a star around  $R_g Q \approx 1$ .

In order to prove this interpretation experiments on partially labelled star molecules were performed. Fig. 75 displays the reduced relaxation rates ( $\Gamma/Q^3$ ) for a protonated 12-arm PS-star in deuterated THF. Furthermore it shows the results obtained from a deuterated star, where one arm was labelled (protonated). In the latter case the deuterated THF matched the deuterated parts of the star molecules and only the dynamic response from the labelled arm was seen. The difference between the two results is striking. While for the fully labelled star the reduced relaxation rates  $\Gamma/Q^3$  exhibit the expected minimum, the single arm rates cross over smoothly from internal Zimm dynamics at large  $Q$  to translational diffusion at low  $Q$  (in this plot  $\Gamma \sim DQ^2$  leads to a  $1/Q$  dependence). Obviously the minimum is a signature of the collective response of the many arm system as inferred from the earlier experiment.

### Structure investigation of model microgels

Macromolecular  $\mu$ -networks with different molecular weights and crosslinking densities have been examined by SANS (Mainz, ILL). As a model for macroscopic networks these structures show in dependence of the environment (good solvent,  $\Theta$ -solvent, linear and crosslinked bulk-matrix) an increase of the exponent of the Fourier transform of the spatial pair-correlation function, compared to linear chains. This behaviour can be described with a scaling law and corresponds to a "fractal" molecular architecture. With decreasing solvent quality and increasing sterical hindrance the  $\mu$ -networks become more compact in a well characterizable way thus gaining possibly a new insight into the behaviour of crosslinked materials.

### Deformed polymer mixtures

Recently, SANS experiments have been conducted on mixtures of polystyrene and poly(phenylene oxide) deformed below  $T_g$  (Mainz, ILL). The aim of these experiments is to study the effect of high stresses and strains on concentration fluctuations in polymer mixtures (see fig. 76).



Figure 76: SANS from concentration fluctuations in a mixture of Polystyrene and Poly(phenylene oxide), deformed below  $T_g$  (stretching direction horizontal).

### Strained networks

The SANS study of labelled free chains in a strained network (Strasbourg, Mainz) gave rise to an unexpected scattering pattern, which was baptized "butterfly pattern" because of its characteristic shape (see fig. 77). The example shown here is an endlinked PDMS network with an average meshsize of 10000 m.w. This was "swollen" with 14 % of smaller deuterated PDMS oligomers ( $M=6000$ ) and stretched to  $\lambda=1.3$ . Note that the usual elliptic scattering patterns are oriented perpendicularly with respect to the drawing direction! This reversible effect can eventually be understood in terms of a spinodal decomposition, which occurs under strain and therefore shows this striking anisotropy.

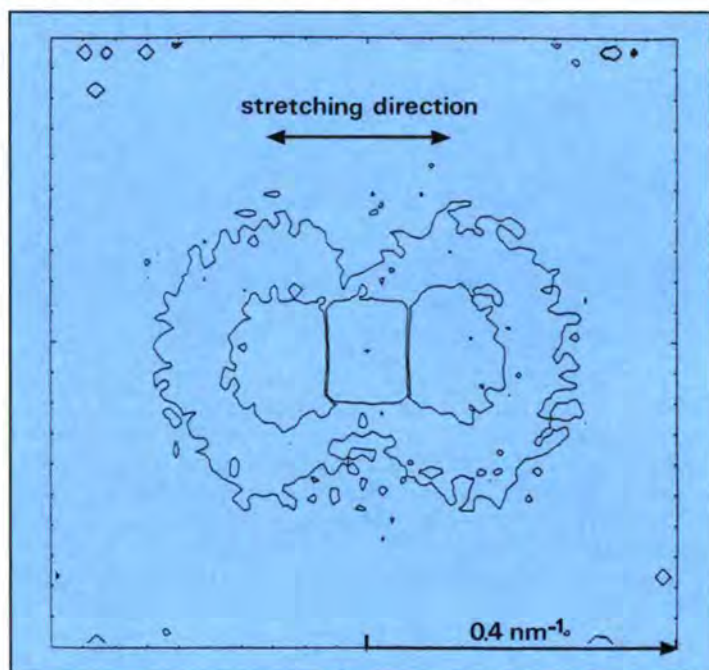


Figure 77: SANS-"butterfly"-pattern of labelled free chains in a strained PDMS network (stretching direction horizontal).

### Coalescence in latex films

Looking on coalescence of latex films, SANS of a n-butylmethacrylate system revealed a strong interdiffusion of molecules of neighbouring latex particles. This interdiffusion is found to be strongly dependent on molecular mass, compatibility of the polymer material and annealing temperature (Ludwigshafen, ILL).

### Drag reduction

A continued project is the SANS investigation at D11 of surfactant solutions, containing rod-like micelles, under laminar and turbulent flow-conditions (Dortmund, ILL). At lowest concentrations surfactant-additives (as well as high molecular weight polymers) behave as drag reducers, i.e. the friction factor in a turbulent flow at a constant flow rate is reduced. Although this effect has greatest importance with respect to technological applications (e.g. savings in pumping energy) the structural changes under shear on a molecular scale are still under discussion. Results of recent SANS-experiments in a pipe-geometry in turbulent flow and comparison with shear-experiments in laminar Couette flow, static SANS-investigation of the solution at rest and static and dynamic light scattering

experiments (in collaboration with the EMBL-outstation, Grenoble) seem to indicate that the increased local stresses in turbulent flow partially breaks up the micellar structure, thus leading to a broader distribution of the particle sizes and a decrease of the average rod length. The micellar rods are however completely aligned in the flow direction; in the investigated Q-range an indication of an intermicellar ordering was not observed in the scattering pattern.

### SANS with high pressure equipment

In SANS-studies of physical chemistry-processes, pressure remained so far the least used of the common experimental variables. Recent advances in high pressure studies were due to improved instrumentation and the more general availability of high pressure equipment. A high pressure cell, designed for pressures up to 40MPa, has been built (Osnabrück) especially for use with neutron small angle scattering. This cell has successfully been applied to evaluate the phase diagrams of lipid mixtures (Mainz, ILL) and to study the influence of high hydrostatic pressure on the conformation of polyelectrolytes in aqueous solution (Osnabrück, ILL).

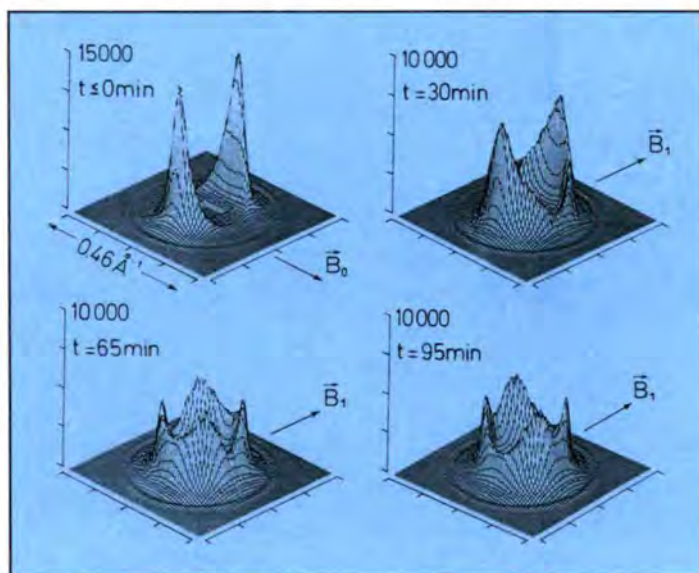


Figure 78: Scattering intensity as a function of time of a nematic sample of rodlike micelles, prealigned in a magnetic field  $\vec{B}_0 = 5\text{ T}$  and exposed to a field  $\vec{B}_1 = 1.4\text{ T}$  (perpendicular to  $\vec{B}_0$ ) during the scattering experiment.

### Neutron scattering of nematic rodlike micelles in a magnetic field

In a real time experiment the magnetic orientation of rodlike micelles was studied with SANS at D17 (Bayreuth, ILL). A nematic sample of cetyltrimethylammoniumbromide (CTAB) in  $\text{D}_2\text{O}$ , prealigned in a magnetic field at 5 T, was positioned between the poles of a second magnet of field  $\vec{B}_1 = 1.4\text{ T}$  in the neutron beam. The direction of the neutron beam, the director of the nematic phase  $\vec{D}$  and the direction of  $\vec{B}_1$  were perpendicular to each other. When the field  $\vec{B}_1 = 1.4\text{ T}$  was switched on at time  $t=0$ , the director of the nematic sample slowly turned clockwise and counterclockwise into the direction of  $\vec{B}_1$ , as can be seen from the SANS pattern measured as a function of time (see fig. 78). Conclusions of these experiments are: the nematic phase consists of rodlike micelles and the rods align with their axis parallel to the magnetic field.

The realignment in the given field of 1.4 T is slow (time scale of hours), a reason for that being the high viscosity of the nematic phase. Additional birefringence measurements give evidence that the relaxation of the nematic director proceeds in a cooperative way in which whole domains are rotated clockwise and counterclockwise.

### SANS of sheared concentrated dispersions

Polymer lattices can be prepared by various techniques as dispersions which contain spherical particles with a very narrow size distribution. SANS provides a means to probe the microstructure of such concentrated colloidal suspensions during simple shear flow. Dispersions of charged, spherical polystyrene latex particles are strongly interacting and form interesting model systems for the study of long range particle-particle interactions. The diffraction pattern of such an aqueous based latex of diameter  $0.2 \mu\text{m}$ , observed at D11 at 37m is shown in fig. 79. The experiment was carried out in the ILL Couette shear cell and the application of the shear gradient produced a highly ordered "crystalline" arrangement of the particles. Subjected to a range of shear rates up to  $10^4 \text{ s}^{-1}$  a number of changes in the diffraction pattern (see fig. 80) were observed (Bristol, ILL).

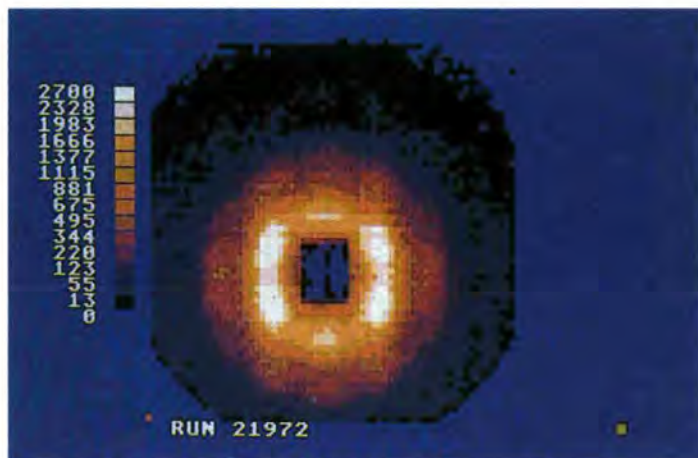


Figure 79: Diffraction peaks from an ion-exchanged polystyrene latex at D11.

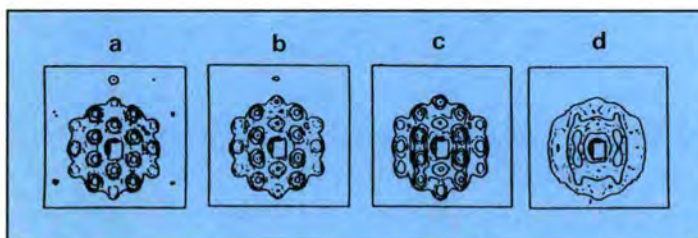


Figure 80: Effect of shear on polystyrene latex structure a) zero, b)  $10 \text{ s}^{-1}$ , c)  $1600 \text{ s}^{-1}$ , d)  $10^4 \text{ s}^{-1}$

A system with short range particle-particle interactions is poly(methyl methacrylate), stabilised by an outer layer of poly(12-hydroxy stearic acid) and dispersed in dodecane at a concentration of 44% w/w. Fig. 81 shows the results of a shear experiment at D11 in the range of shear gradient up to  $6000 \text{ s}^{-1}$ . With increasing shear gradient significant changes of the scattering pattern are observed and the results suggest that under sheared conditions the particles are forming chains along the direction of flow (Bristol, ILL).

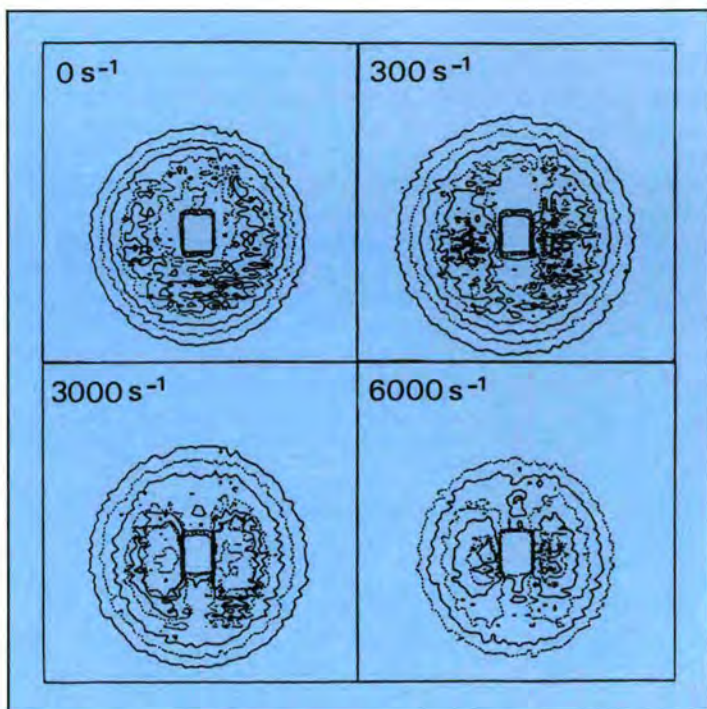


Figure 81: Effect of shear on a poly(methyl methacrylate) latex; a) zero, b)  $300 \text{ s}^{-1}$ , c)  $3000 \text{ s}^{-1}$ , d)  $6000 \text{ s}^{-1}$

Another system that interacts according to the hard sphere potential and mimics the behaviour of simple molecular liquids, are sterically stabilized, colloidal silica spheres, suspended in cyclohexane. Suspensions of this type were investigated at D11 in a Couette type shear-cell built at Utrecht. The equilibrium structure of the suspension is similarly disturbed by the flow and the resulting anisotropy is reflected in fig. 82 which shows the ratio of the scattering pattern during shearing to that of the sample at rest (Utrecht, ILL). The data are used to determine the structure factor of the suspension and to evaluate flow-induced structural changes. These structural changes correlate with the rheological behaviour of the suspensions.

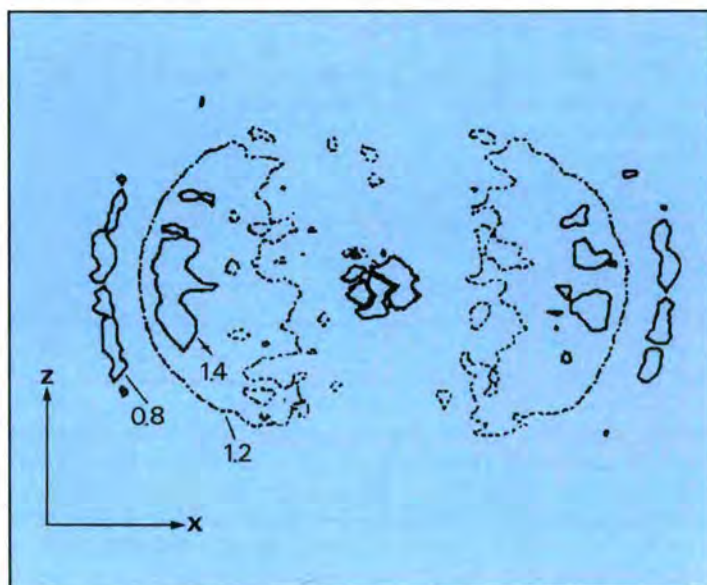


Figure 82: Ratio of scattering patterns,  $I_x = 4000 \text{ s}^{-1} / I_x = 0$ , for colloidal silica particles suspended in cyclohexane at a volume fraction of 0.523. Simple shear flow is given by  $v_x = \gamma(0,1,0)$

## **Microemulsions**

Experiments on the D16 and D17 spectrometers have permitted to progress in the understanding of the structural properties of microemulsions, particularly when they are bicontinuous. By choosing appropriate combinations of deuterated components it has been possible to obtain the scattering from the surfactant layer. From this it has been shown that the amphiphilic film in the bicontinuous microemulsions forms infinite periodic minimal surfaces (Paris, ILL).

## **Training course in small angle neutron scattering**

The ILL has always been trying to encourage new users of its facilities. It is aware of the fact that the short duration of a scheduled experiment is insufficient for becoming familiar with all the features of a complicated instrument and its most efficient operation. Small angle scattering experiments on colloids and polymers have an average time allocation of less than two days. To alleviate the difficulties, a short training course was organized for students and post-docs interested in using small angle neutron scattering (SANS) in problems concerning polymers and colloids. About a dozen participants came to the ILL for two and a half days in March. The course consisted of a short theoretical introduction, demonstration of the SANS instruments and a practical session during which both standard and the students' own samples were measured and evaluated. We believe that this course proved useful; it was perhaps too short to yield the full benefit expected. Since we have at the ILL a considerable number of scientists working in the field of SANS it is hoped that the course can be repeated in the near future on a broader basis and in a modified form.

Secretaries: H. Blank (9a)  
P. Lindner (9b)

## Polymer Motion in Dense Systems

Diffusive motion in dense polymer systems ranges from large scale Brownian motion in entangled melts which may occur on macroscopic time scales to fast small amplitude fluctuations in the neighbourhood of the glass transition. To a large extent these intramolecular relaxations determine the viscoelastic and transport properties of polymeric materials which govern many of their applications. Their investigation has been a long standing goal of polymer science and has attracted scientists applying a broad spectrum of different experimental tools. In particular a variety of microscopic methods has been developed revealing detailed information on the microscopic nature of these dynamic properties. In order to bring together scientists working in this field and to compare the results obtained with different techniques, in collaboration with the Institut für Festkörperforschung of the KFA Jülich, the ILL has organised a workshop on "Polymer Motion in Dense Systems" devoted to their microscopic aspects. It took place in September 1987 at the ILL. Here, I briefly review the range of discussion and present selected examples [1/.

The first session dealt with local relaxation processes in bulk polymers. They are accessed by a variety of techniques: Fluorescence anisotropy decay measures the time dependent segment orientation auto-correlation function and provides information on the single bond dynamics. The requirement of fluorescent labels, however, may cause important local disturbances. Photocorrelation techniques and classical NMR reveal frequency distributions of the relaxing species but very limited spatial information. Quasielastic neutron scattering in principle elucidates the space time correlation functions on an atomistic scale but is limited to relatively fast processes  $\tau_c \leq 10^{-7}$ s. Here, the so-called 2-dimensional NMR-methods have evolved as a new tool measuring directly the orientational part of the van Hove correlation function and revealing the space time behaviour of the local relaxation process in a time window inaccessible to neutrons ( $10^{-6} \leq \tau_c \leq 10^2$  s) [2/]. In such a deuteron NMR experiment a sequence of three high frequency pulses is applied (Fig. 83). The first two pulses are a reminiscence of the usual NMR spin echo set up which creates the "solid echo" after a time  $t_m = t_1$ . The dynamics of slow motion with correlation times in a range of  $10^{-6} < \tau_c < 10^{-3}$  s can be analyzed by studying the solid echo lineshapes as a function of  $t_1$ . In the case of nuclei with quadrupole moments like the  $^2\text{H}$  the second pulse also creates a particular state of quadrupolar ordering with respect to the axis of the main field. During the mixing period  $t_m$  after the second pulse, this order can be altered due to spin lattice relaxation (normally negligible) or by reorientational processes. The third pulse flips the remainder of this quadrupolar order into the perpendicular plane and arises the stimulated or alignment echo

$$S(t_1, t_2) = \langle \exp[i\omega(o)t_1] \exp[-i\omega(t_m)t_2] \rangle \quad (1)$$

The frequencies  $\omega(o)$  and  $\omega(t_m)$  are the NMR frequencies at the beginning and the end of the mixing period; the brackets denote the thermal average.

For  $t_1 = t_2$  Equ(1) is formally identical to the expression for the intermediate scattering function for incoherent neutron scattering

$$I(Q, t) = \langle \exp(i\vec{Q} \cdot \vec{r}(o)) \exp(-i\vec{Q} \cdot \vec{r}(t)) \rangle \quad (2)$$

if the momentum transfer  $Q$  is replaced by  $t_1$  and the position vector  $\vec{r}$  by  $\omega$ . Since the NMR frequency  $\omega$  is characteristic for the molecular orientation  $S(t_1, t_2)$  measures the orientational part of the self-correlation function observed in neutron scattering. However, the NMR time scale of  $10^{-6} - 100$  s is very different from the neutron time scale  $t < 10^{-7}$  s and complementary information can be obtained. In the general case  $t_1 \neq t_2$  the off diagonal part reveals cross correlations between NMR frequencies or molecular orientations before and after the evolution time. Fig. 84 presents the results of such a measurement on main chain deuterated PS in the vicinity of the glass transition  $T \geq T_g = 100$  C. Fig. 84 shows the Fourier transform of  $S(t_1, t_2)$ . In the diagonal direction ( $t_1 \times t_2$ ) a strongly broadened structure is visualised. It is indicative for a diffusional process, where during each step a chain segment turns only by an infinitesimal amount. The pronounced off diagonal signals demonstrate that in addition during the mixing time  $t_m$  certain chain segments have lost their orientational correlations indicating larger conformational rearrangements. These first experiments on polymer dynamics are very promising and introduce 2-D NMR as a serious contender to quasielastic neutron scattering in unravelling space time correlations of local reorientational motions.

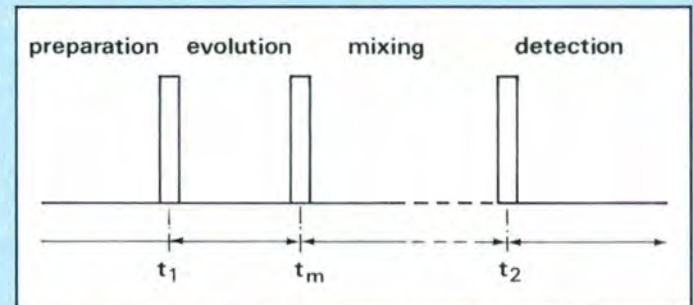


Figure 83: Typical pulse sequence for 1-D and 2-D NMR experiments

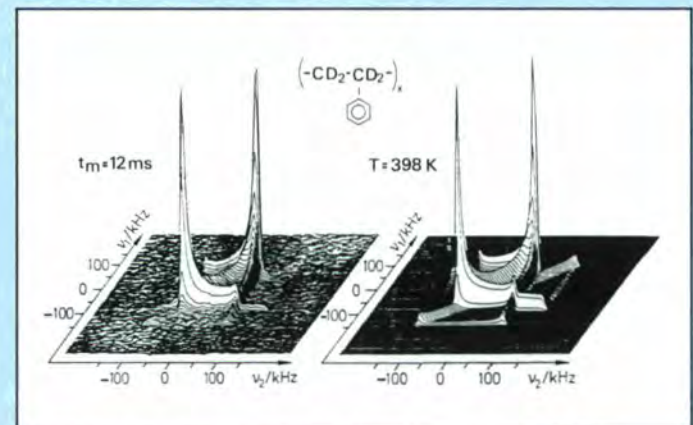


Figure 84:  $^2\text{H}$ -2D-FT-NMR spectrum of chain deuterated polystyrene. A superposition of the models of small step rotational diffusion and rotational random jumps was used for the calculated spectrum.

In the second session the time dependent small angle neutron scattering (SANS) approach to the dynamics of polymers was discussed. In such experiments the relaxation from a

non equilibrium state is observed by watching the time dependent scattering pattern. This can be done in a real time experiment, where e.g. SANS patterns of a rubber under periodic stretch are accumulated or by an investigation of a series of samples which were quenched into the glassy state after a certain evolution time. These experiments probe the polymer dynamics on macroscopic time scales and are in particular suited to investigate long range chain motions such as reptational diffusion in the melt. As an example I discuss an experiment aiming on the so called tube disengagement process in the reptation model /3/. The reptation model /4/ describes the topological constraints imposed on a given chain by the presence of the other chains by a tube along the chain profile. Significant chain motion is only possible within the tube. As shown schematically in Fig. 85 for long times the chain will disengage from its initial tube by its extremities. This idea was tested in a SANS experiment on triblock PS-chains which were labelled either in the center or at the extremities and dissolved in a high molecular weight PS matrix ( $M_w = 10^6$ ). The samples were uniaxially stretched ( $\lambda = 3$ ) at  $140^\circ\text{C}$  and after holding at fixed strain for times  $t_r = 100, 300, 1000\text{s}$  quickly quenched to room temperature. Fig. 86 presents the time evolution of parallel and transverse components of the scattering function in a Kratky representation ( $I(Q) Q^2$  vs  $Q$ ). The equilibrium state is the isotropic pattern. From these plots it is clear that the endlabelled chains recover the equilibrium conformation much faster than the chains labelled in the center in qualitative agreement with the reptation concept. Rheological experiments performed on the same samples yielded time constants in agreement with the SANS-results.

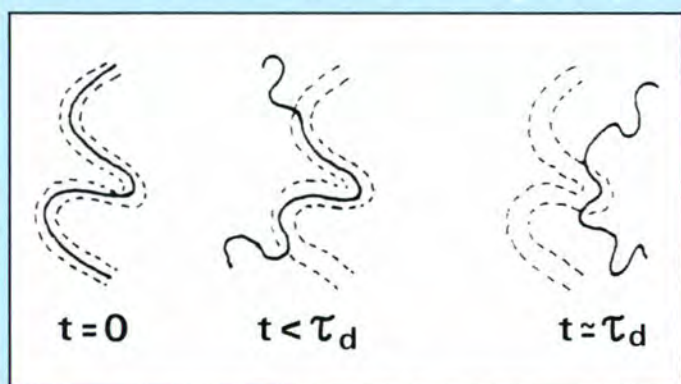


Figure 85: Disengagement of a polymer chain from its initial tube.

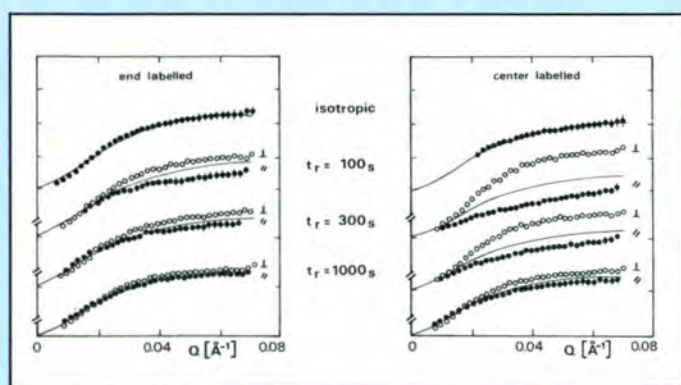


Figure 86: Intermediate range of  $Q$  - Kratky plots of the parallel and transverse components of the intensity scattered by blends of end- and center labelled polystyrenes in a PSH matrix ( $M_w = 10^6$ ).  $t_r$  is the experimental relaxation time.

In the third session fast fluctuations near the glass transition and in the glassy state were discussed. Thereby, fast fluctuating hydrodynamic modes which are observed in photon Brillouin scattering experiments have to be distinguished from the newly discovered ultra fast local motions which appear to accompany the glass transition. These fast local motions have been observed in inorganic, molecular as well as polymer glasses and were interpreted either by a transfer of vibrational density of states towards low frequencies due to a general softening of the structure /5,6/ or by aperiodic relaxational processes /7/. Their existence has been predicted by mode coupling theories of the glass transition. These theories also account for the observed temperature dependence of the fluctuation amplitudes /8/.

In the fourth session the large scale Brownian motion of long chain molecules and their diffusion in an entangled environment was considered in the light of new experimental results from techniques including neutron spin echo and forced Ragleigh scattering. Besides analytic theories like the reptation model in this field computer simulations are an important guidance for the interpretation of experimental results. Simulations on polymer motion under entanglement constraints or through fixed obstacles were presented. A new cooperative motional mechanism on the basis of closed loop rearrangements was also proposed. While computer simulations in general reproduce the predictions for the macroscopic dynamics of the reptation model: i.e.  $D \sim N^{-2}$  and terminal time  $\tau_t \sim N^3$ , where  $N$  is the number of monomers along the chain none of the simulations has clear evidence for the microscopic prediction of a mean square segment displacement  $\langle \Delta r^2(t) \rangle \sim t^{1/4}$  but rather always the Rouse type  $t^{1/2}$ -law is found. So far experimental results by neutron spin echo are ambiguous: while the  $Q$ -dependence of the characteristic frequency  $\Omega$  follows the Rouse prediction  $\Omega \sim Q^4$ , the experimental lineshape obtained from coherent scattering appears to be in better agreement with the model of local reptation. In order to gain further insight on the role of topological constraints in the microscopic dynamics of polymer melts, a new set of experiments on PDMS chains under varying constraints was performed /9/. Thereby labelled PDMS-chains ( $M_w = 100000$ ) were trapped in a deuterated network of narrow mesh sizes, in order to simulate more severe hindrance or a narrower tube.

The NSE-experiments were performed at  $100^\circ\text{C}$  under the same conditions as earlier measurements on the melts /10/. Figure 87 compares the lineshape parameters obtained from the different samples starting from the concentrated solution up to the small mesh size network. Within the experimental error for all samples  $n = 1/2$  is found (for the small mesh size sample the statistics do not suffice to obtain  $n$  for each spectrum individually). Figure 88 compares the characteristic frequencies obtained from the same set of samples. They were determined by fitting the coherent scattering function of the Rouse model to the respective spectra. The solid lines display the  $\Omega \sim Q^4$  scaling law of the Rouse model. In all cases this relationship is followed very closely by the data. We realize further that with increasing constraints the curves are shifted towards lower rates indicating a reduction of the Rouse parameter with increasing hindrance. In going from the concentrated solution to the trapped chain in a network with only about 40

monomers between crosslinks the entanglement distance was changed by a factor of 2.5. Despite this important change of the constraints acting on a chain the experimental data do not change their characteristics neither with respect to the lineshape nor with respect to the Q-scaling of the characteristic frequency. The latter is in particular puzzling since an approach to local reptation in the sense of De Gennes would have meant an increase in the power law: At high Q a more or less undisturbed Rouse relaxation is expected while towards smaller Q's the constraints are supposed to become increasingly effective yielding a stronger reduction of the characteristic frequency than due to Rouse motion alone. Instead one observes an increasing reduction of the Rouse parameter with increasing constraints. While the difference between the concentrated solution and the melt is easily attributed to a changing segmental friction coefficient due to the addition solvent, in the melt and the networks the friction coefficient being a local property should not change. The observation of an overall reduction of the chain mobility rather seems to indicate that the increase of constraints is active on a range of length scales yielding a general reduction of the time scale of motion. The prevailing Rouse characteristics independent of the amount of topological constraints agree well with the computer simulations mentioned earlier /11/.

Important progress has been achieved in unravelling the characteristics of phase separation in polymer blends. The modifications to the ordinary laws of diffusion are of particular interest. In this field new time dependent neutron and light scattering technique have played a significant role. In particular large extensions of the spatial resolution in neutron scattering lead to new results. As an example I briefly discuss an experiment on the growth of precipitates in the miscibility gap of the polymer blend PVME-PS/12/. There time dependent scattering pattern have been obtained in real time and experimental results from ordinary small angle scattering were combined with data from a neutron Bonse-Hard camera covering a Q-range of more than four orders of magnitude. Fig. 89 presents scattering patterns after annealing times of 4 and 20 min at 146°C which is slightly above the critical temperature of 142°C. At small Q ( $Q < 2.5 \cdot 10^{-4} \text{ \AA}^{-1}$ ) the scattering profile follows a Guinier formula revealing the radius of gyration of the domains and extrapolating to  $Q \rightarrow 0$  their volume. At intermediate Q's ( $6 \cdot 10^{-4} < Q < 2 \cdot 10^{-3}$ ) a Porod law is found. Towards higher Q the shape increases to  $Q^{-6}$  indicating a diffuse domain boundary of about 400 Å thickness. Finally at large Q critical scattering from inside the domains is seen. Fig. 90 presents the obtained growth laws for the volume of the precipitating domains. At short times the increase in the domain size  $R \sim V^{1/3}$  is compatible with a  $t^{1/3}$  law characteristic for growth by diffusion and agglomeration, the later linear growth is interpreted by hydrodynamic effects.

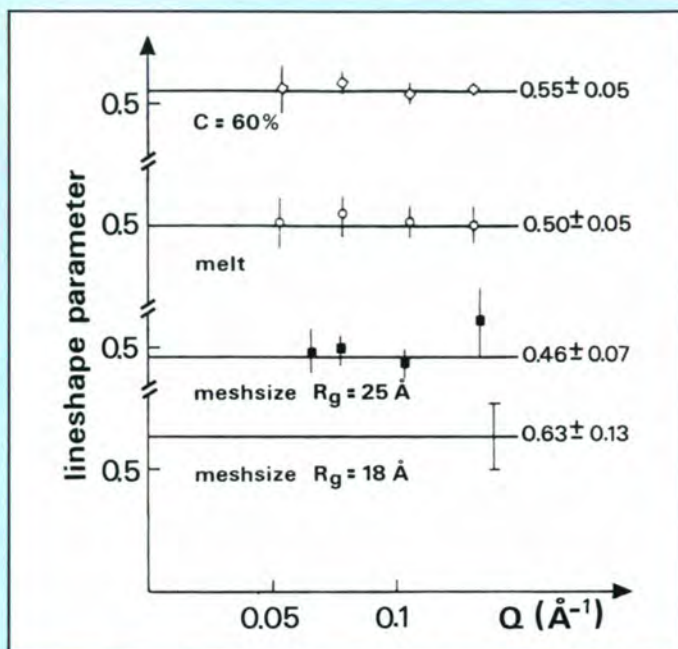


Figure 87: Comparison of the lineshape parameters ( $S(Q,t) \sim \exp(-Q^2(t/\tau_c)^\beta)$ ) obtained from a concentrated solution ( $M_w = 178000$ ; monomer conc. 60%), the PDMS melt ( $M_w = 60000$ ) and PDMS chains ( $M_w = 100000$ ) trapped in networks of different mesh sizes. All measurements were performed at 100°C. The solid lines are the result of respective joint fits to the data.

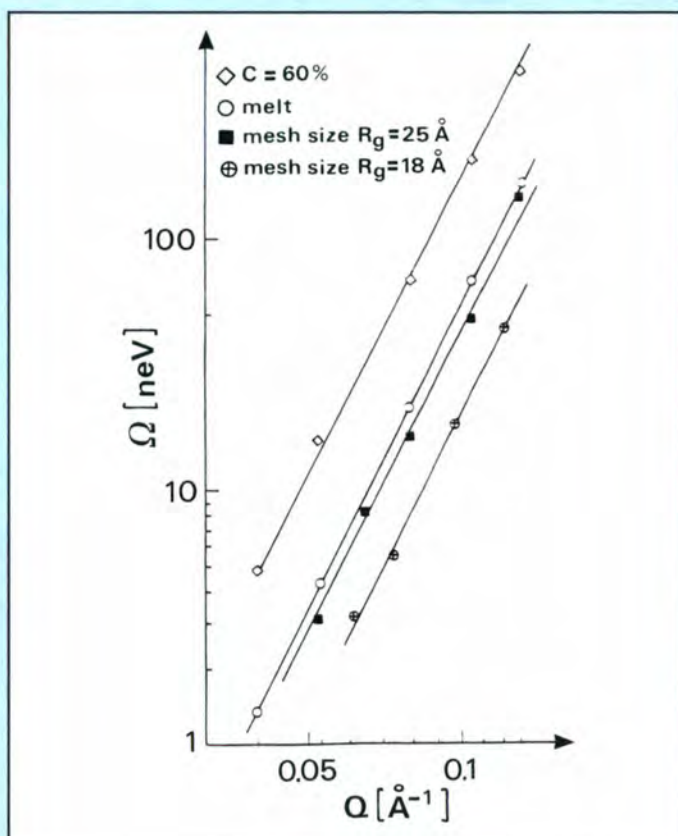


Figure 88: Characteristic frequencies for the concentrated solution, the PDMS melt and the trapped chains. The solid lines display the  $\Omega \sim Q^4$  scaling law.

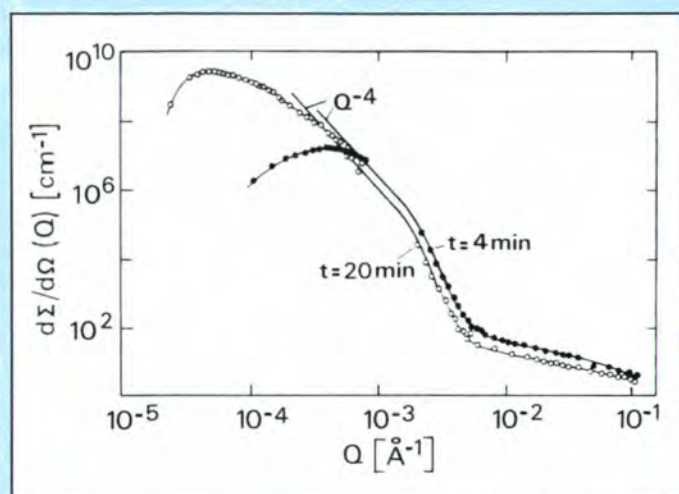


Figure 89: Two selected scattering patterns for  $t = 4$  and  $20$  min at  $146^\circ\text{C}$  as measured by the double crystal diffractometer and the conventional pinhole small angle scattering instrument, for a blend with  $M_w(\text{PVME}) = 89.000$  and  $M_w(\text{d-PS}) = 232.000$  at critical composition. The critical temperature is  $142^\circ\text{C}$ . Scattering on the domains, surface (Porod) scattering, and the scattering due to fluctuations inside the domains can be seen.

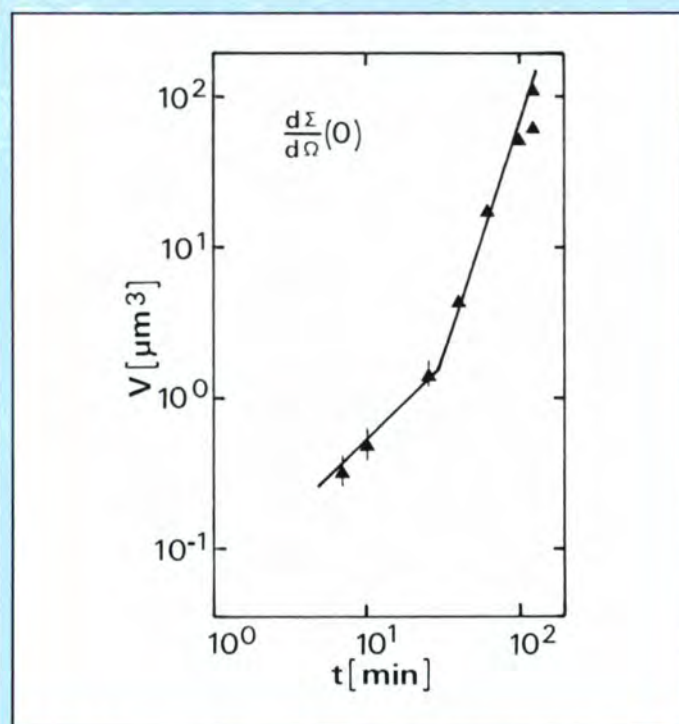


Figure 90: Characteristic volumes  $V = (4\pi/3) R^3$  ( $R$  = characteristic length) from  $d\Sigma/d\Omega(Q=0)$ . The solid curves, corresponding to  $t$  and  $t^2$  power laws, are guide lines for the eye.

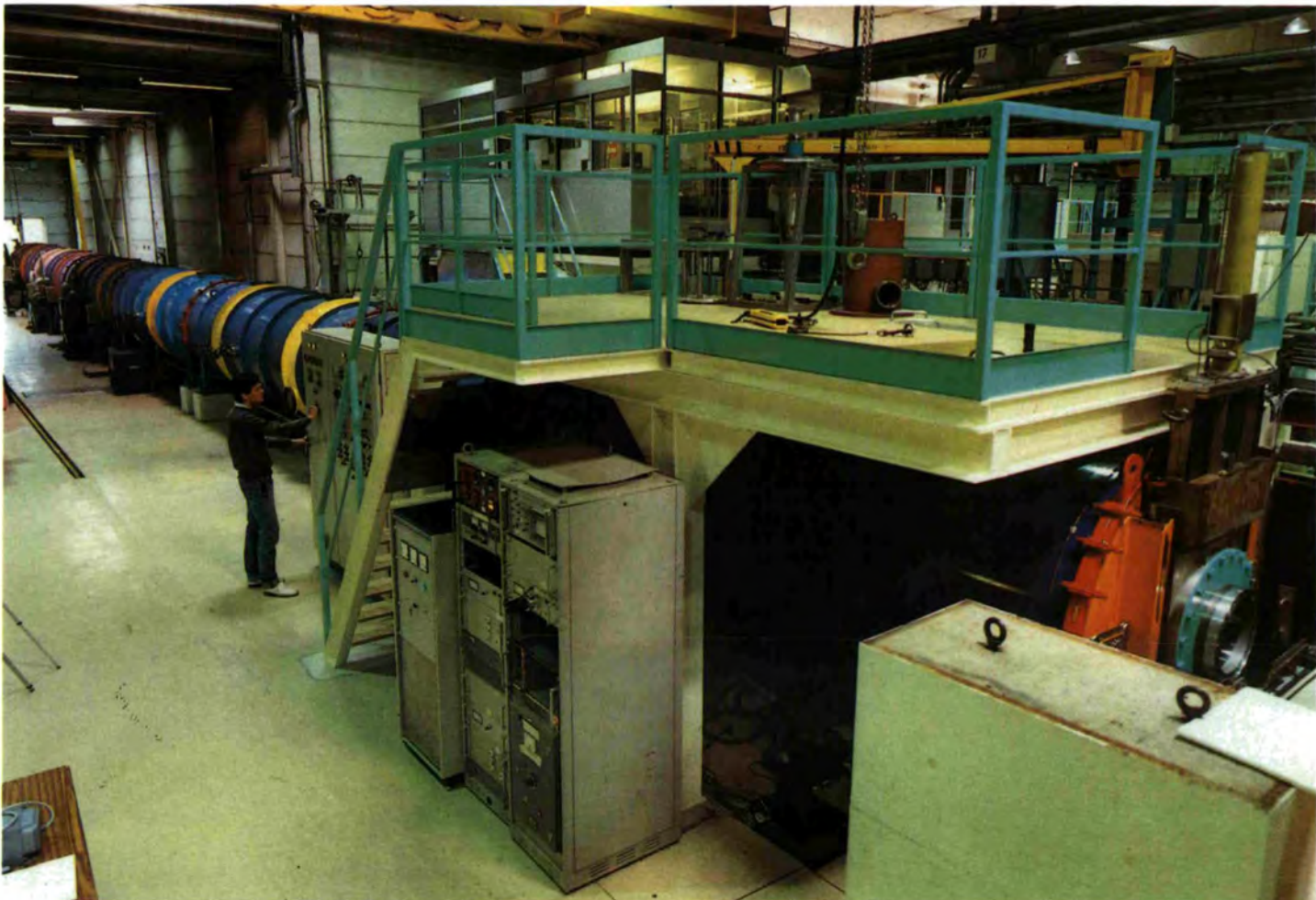
I hope that the workshop having dealt with polymer motion near the glass transition, in entangled systems and during phase separation in polymer blend, may stimulate further studies. In particular, a better understanding of microscopic motion should provide a bridge to macroscopic rheology. Further perspectives will also strongly depend on new developments in instrumentation. Dedicated small angle scattering machines for time dependent studies at high flux reactors will increase the achievable time resolution of real time experiments significantly. Neutron spin echo methods using advanced magnet field design in combination with

long wavelength neutrons will increase the space time domain accessible to neutron scattering by more than an order of magnitude. Synchrotron radiation will revolutionize X-ray scattering. Multi-pulse techniques in NMR are on the way to greatly extend the accessible time range and will provide information complementary to that obtained from scattering methods.

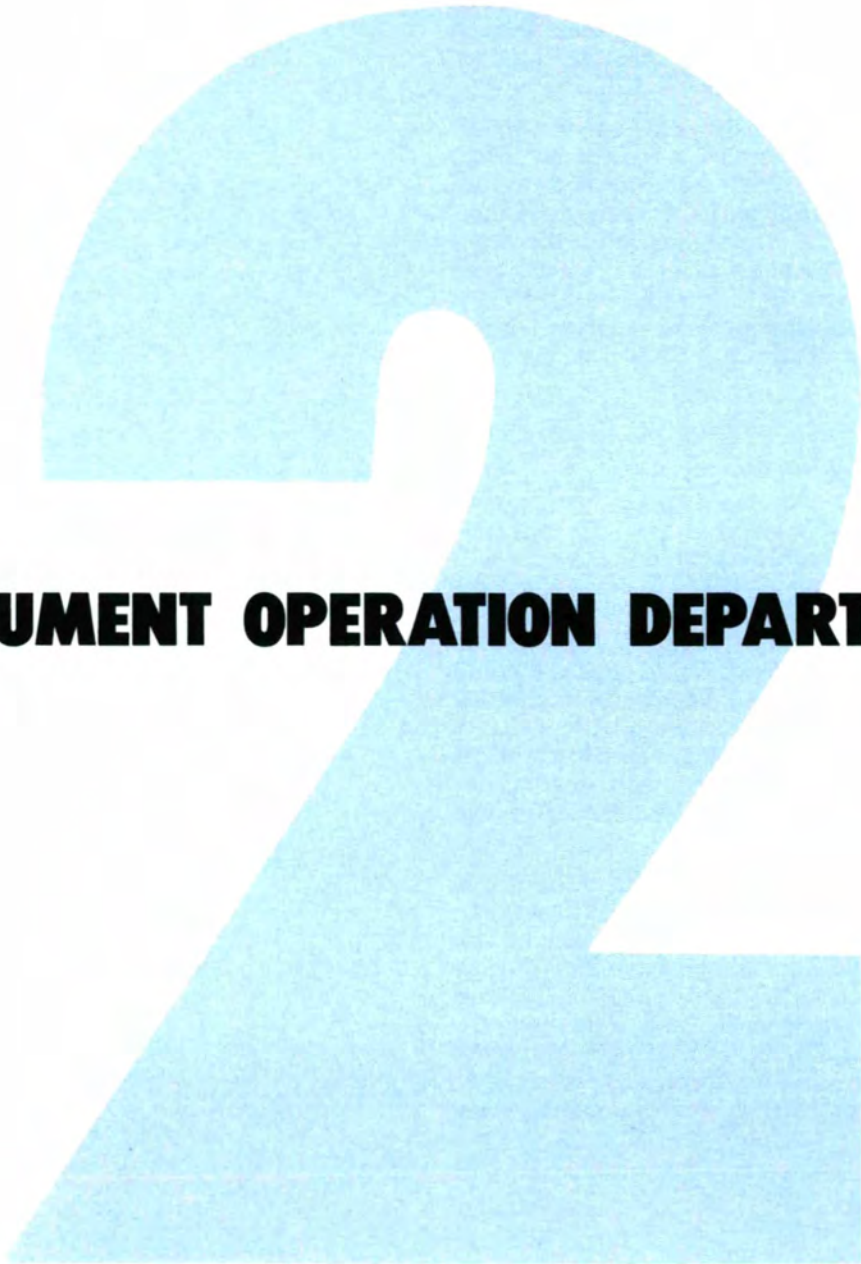
D. Richter

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*D11, the small angle scattering facility on the H15 guide.*



**INSTRUMENT OPERATION DEPARTMENT**

## **EINLEITUNG**

Die Abteilung Instrumentenbetrieb wurde 1987 mit neuen Problemen und Herausforderungen konfrontiert. Ein Problem war die Installation von fünf neuen Instrumenten an der Horizontalen Kalten Quelle bei gleichzeitiger Reduzierung der Anzahl der Mitarbeiter, eine vom ILL eingegangene Verpflichtung nach Beendigung des "deuxième souffle". Eine weitere Herausforderung war, Ideen für eine fruchtbare Zusammenarbeit zwischen ILL und ESRF vorzuschlagen. Es wurden zwei Arbeitsgruppen gegründet, um Möglichkeiten für eine gemeinsame Gelände-Verwaltung sowie gemeinsame Einrichtungen für eine Benutzerunterstützung zu untersuchen.

Die Hauptaufgabe des Personals in der Abteilung Instrumentenbetrieb besteht darin, Wissenschaftler bei der Durchführung ihrer Experimente zu unterstützen. Aber dennoch wurden 1987 in denjenigen Bereichen einige Entwicklungen vorangetrieben, in denen es schwierig oder unmöglich ist, das benötigte Gerät zu kaufen. Beispiele einiger Projekte sind: Verflüssigungskryostat-Einsatz für einen existierenden 6-Tesla-Kryomagnet; 5mK Verflüssigungskryostaten mit hoher Kühlleistung; Tieftemperatur-Kryostat (2 K) für Vierkreis-Diffraktometer.

## **INTRODUCTION**

Le Département EDEX a été confronté en 1987 à de nouveaux problèmes ainsi qu'à de nouveaux défis. Un des problèmes a été le fait que cinq nouveaux instruments seront installés sur la source froide horizontale et que le personnel doit être réduit pour s'acquitter de l'engagement de l'ILL pour la période d'après le "deuxième souffle". On lui demande de proposer des idées pour une coopération fructueuse entre l'ILL et l'ESRF, ce qui représente un défi. Deux groupes de travail ont été créés pour étudier les possibilités d'une administration commune du site et d'installations communes comme support pour les utilisateurs.

La tâche principale du personnel du Département EDEX est de fournir aux scientifiques un support pour effectuer leurs expériences. Mais quelques travaux de développement ont également été faits en 1987 dans des domaines où il est difficile ou même impossible d'acheter le matériel requis. Quelques exemples de projets de développement: insert à dilution pour un cryo-aimant existant de 6 Tesla, un cryostat à dilution de 5 mK à haute capacité de refroidissement, un cryostat (2 K) à basse température pour des diffractomètres à quatre cercles.

## **INTRODUCTION**

The Instrument Operation Department was faced in 1987 with new problems and challenges. One problem resulted from the fact that five new instruments will be installed at the horizontal cold source and that the number of staff has to be reduced in order to fulfil the commitment the ILL has made for the period after the "deuxième souffle". The request to propose ideas for a fruitful cooperation between the ILL and the ESRF is a challenge. Two working parties were set up to study possibilities of a common site administration and of common facilities for user support.

The main task of the staff in the Instrument Operation Department is the provision of support to the scientists performing their experiments. But some development work was also done in 1987 in areas where it is difficult or impossible to buy the required equipment. Examples of some development projects: dilution refrigerator insert for an existing 6 Tesla cryomagnet, 5mK dilution refrigerator with high cooling power, low temperature (2 K) cryostat for four-circle diffractometers.

## **ADVANCED CRYOGENIC SERVICE**

### **THERMOMETRY AND INSTRUMENTATION**

The number of ILL Precision Temperature Controllers in service at the ILL now stands at 39. Six more are due to be delivered in December 1987. Seven others are in use outside the ILL, and the manufacturer is, at the time of writing, in the process of negotiating the right to commercialize them under licence. The same is true for the ILL helium level gauges for cryostats and automatic cold valve systems.

Early in 1988 it is planned to test, in real experiments, new software permitting the control of the sample temperature using a second PID loop. This will have two main advantages: The sample and not the regulation block will be maintained at the set-point temperature. The time taken for the temperature of the sample to reach equilibrium can be decreased by as much as a factor of 2.5.

### **SUPERCONDUCTING MAGNETS**

After last year's change of coils of the 6 Tesla cryomagnet, this has been used almost continuously on the 3-axis instruments (particularly IN20), and the maximum field was regularly achieved without difficulty.

However on two occasions leaks were found on the variable temperature insert. The repair of this leak necessitated the complete dismantling of the cryostat. The design of the defective Indium seal assembly has been improved.

The construction of a dilution insert, designed for this cryomagnet, was started and has made good progress. It is expected that temperatures of the order of 20 mK will be ob-

tained with the 6 Tesla field. Tests are planned for the coming months.

The 7.3 Tesla cryomagnet, for general use, has been equipped with a new power supply system, with a computer-controlled ramp, permitting automatic changing of the magnetic field.

The 7 Tesla cryomagnet, with a room temperature bore, is kept cold permanently, with the magnetic field. It is used for the preparation of samples usually in collaboration with EMBL.

### **VERY LOW TEMPERATURES**

The cryostats developed in previous years are now used for routine operation. The actual number of neutron measuring days at very low temperature ( $T < 1K$ ) was 200, divided between 12 instruments and 30 samples. The high degree of flexibility of this service has been widely utilized by the experimentalists. For example 4 samples were measured in 10 days with the same cryostat on D1A. Another example is the measurement at very low temperature of a sample under high pressure in a clamp cell (30kbar) with a weight of 1kg. Special assistance was given for measurements of He<sup>3</sup>-He<sup>4</sup> mixtures under pressure on IN6.

Another project started 3 years ago, for the construction, testing and adjustment of 3 dilution inserts, has been successfully completed. One insert was supplied to the Rutherford Appleton Laboratory for ISIS, and the two others to NBS Washington. The minimum temperature obtained is less than 20 mK. An additional insert for ILL is under test. It is planned for use in the orange cryostat at 4.2 K without pumping. This can be used as a spare in case of failure of the existing inserts.

A special cryostat "CRYOPAD", for measurements with polarized neutrons, has been designed and built, and is now operational.

A new highly efficient 4-circle cryostat, with continuous liquid helium transfer, has been designed for D10, and its construction is almost complete. Preliminary tests have been successfully carried out. Temperatures down to 1.6 K have been reached with a liquid helium consumption of the order of 0.5 l/h.

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## **CENTRAL SERVICE**

The essential mission of the Central Service is on the one hand the organization of the experimental halls and their management and on the other hand technical assistance on the experiments themselves and on their sample environment.

### **GENERAL INTERVENTIONS GROUP**

The principal tasks of the General Interventions Group are:  
The surveillance of the experimental zones and the satisfac-

tory operation of the experiments as to general and safety facilities.

The surveillance of dangerous samples on the beams, monochromators and shielding.

Tests and checking of all safety features linked to beams and experiments.

Preparation of instructions and management of the alarm systems in liaison with the "salle de contrôle" (SADI).

Maintenance of the fundamental safety state on the experiments.

General management of the halls:

- handling of heavy and varied loads
- maintenance and cleaning - management of gases and cryogenic liquids
- management of shielding material
- organisation of the transport and clearing of snow

Preparation of the general planning for work in the halls and on the experiments, and follow-up.

Management of the dependent laboratories: Laboratory for radioactive samples, chemistry laboratory and X-ray laboratory.

Special effort was needed during the year in particular for transurania experiments on IN8 and D2B, and for a tritium experiment on D10. Modifications were required to the sample handling system to permit the use of the special transurania cryostat on instruments with limited headroom, and to the temperature and pressure sensing devices and alarms.

Work associated with the installation of the Horizontal Cold Source has involved assistance in the dismantling of part of the UCN platform, the dismantling of D5, the transfer of D3, the building of the shielding for the guides in level C and ILL 22, the preparation of the zone for IN14, and the definition of beam shutters and their control system.

Background measurements and the subsequent improvements were made on the shielding of GAMS, IN20, D3B, PN7, IN, S21 and S34.

The small pool of mobile technicians has aided considerably the instruments D19, IN4, DB21 and NN as well as helping to support the "libre service" workshop.

The SADI system is being extended to take into account the instruments to be installed in the new neutron guide hall ILL22.

The campaign to reduce the general level of noise in level C has resulted in a reduction of 3 decibels. This reduction was obtained by fitting all electronic racks with silencers above the extraction fans, reinforcing the rigidity of their side panels and by the installation of fixed pumping systems in acoustic cabinets. The problem of reducing the noise on the mobile pumping system is being studied.

## TEMPERATURE, PRESSURE AND VACUUM GROUP

### Standard Cryostats

New cryostats have been commissioned for D1B and D11/D17. With the installation of D3B, a new cryoflipper was designed, built and tested together with important modifications to the liquid He and N<sub>2</sub> transfer lines. A 2nd generation design cryo-furnace was accepted and component parts are now being manufactured.

Routine maintenance and tests have been carried out on over 40 cryostats, including the thermalisation of thermometers. Routine maintenance on helium and nitrogen vases has increased due to aging of material. Two new 250 l helium vases and two nitrogen vases have been added to the existing stock.

### Vacuum

During the year the inventory of existing equipment has continued, involving the repair and servicing of over a hundred

pumps and ancillary equipment including 20 vacuum gauges. 15 helium pumping groups have been modified and 10 single stage pumps have been transformed into 2 stage units. The number of root pumps has been increased and new pumping groups have been built. Routine maintenance and leak testing has increased.

The pumping station for the new horizontal cold source was completed in the early part of the year and verification and testing continued for several months.

### High Temperature

Over the last two years the high temperature equipment has been very substantially modernized. The group has now a range of 10 complete units (furnace and power console) built or rebuilt over this period. These units are much easier to install than their predecessors as well as being more reliable and more precise. The result is a better service to the scientists, with less beam-time taken up in the preparation.

During 1987, 32 high temperature experiments were performed covering 8250 operating hours, a reduction of 10 % over the previous year. A number of special requests are worth noting: rotating sample mount for IN3 at 1800 K, increased sample zone for an especially large sample on IN1, and a matched pair of furnaces for temperatures from 300 K to 600 K. A new furnace for IN5 is under construction.

The group has expanded its range of refractory materials held in stock for its own maintenance purposes and as a service to scientists.



High pressure BeCu cell for studies under magnetic fields.

### High Pressure

About 20 experiments involving high pressure environment have been performed in 1987 on all types of instruments except small angle scattering instruments. High pressure experiments with pressures up to 5 kbar are now made easier due to the "standardized" pressure cells.

A new version for high temperature is now available. The cell body is made of CuBe. The maximum pressure has been set to 3kbar in order to keep the wall thickness as small as possible and reduce absorption.

Clamped  $\text{Al}_2\text{O}_3$  cells have proved to be unreliable and greater efforts will be devoted to improving the ultimate pressure limit as well as reproducibility.

Experiments have been done at very high pressure ( $P = 24$  kbar) under applied magnetic field. The cell, as shown

in the picture, is made of CuBe with non-magnetic Tungsten Carbide pistons.

It is worth noting that no experiment has made use of the uniaxial stress device.

## BUILDING MAINTENANCE AND MODIFICATIONS SERVICE

The group comprises 25 persons and its main responsibilities are the maintenance and improvement of the ILL site and buildings, and the preparation and adaptation of the experimental positions to the requirements of the users. The group has three essential activities in two main directions.

### MAINTENANCE, REPAIR AND RENOVATION

#### Site and buildings

The main purpose is to keep in an operational state all the buildings and technical installations (electricity, water, drainage, gas, heating, air conditioning, ventilation, handling) except those of the reactor. The group also supervises the general cleaning of buildings, open spaces and work clothes, and it takes charge of waste disposal and sanitary linen. It also carries out the majority of office and laboratory removals and furniture purchases.

#### Experimental positions

In collaboration with the Central Service the group executes all work necessary for keeping in order technical installations (electricity, heating, air conditioning, ventilation, special handling systems) associated with the experiments.

### CONSTRUCTION AND EQUIPMENT WORK

#### Site and buildings

At the request of the Directors or in order to improve the running of existing installations, the group designs and follows up the construction or modification of buildings, offices and

laboratories, and technical installations. The principal work in 1987 has included construction of a building for reactor material storage, restoration of the ILL4 building, re-waterproofing the roof of the ILL3 building, finishing the ILL6 extension and associated installations for the new horizontal cold source compressor, and realization of a fire protection for critical reactor zones in the ILL4 building.

### Experimental Positions

In collaboration with the Central Service, the Reactor Department, and the Instruments and Methods Department, the group defines and organizes the construction and assembly of the biological shielding and infrastructure required around the instruments in the experimental halls. Main projects carried out in 1987 were finishing of the building and infrastructure for the NN-experiment, prefabrication and installation of the major part of the shielding for the new cold source and associated guides, and preliminary work for the installation of D3B and IN14.

### TECHNICAL ASSISTANCE TO OTHER GROUPS

#### General Services

The "Libre Service" workshop and the main sheet metal workshop made possible modifications and production of items which were difficult to sub-contract or were needed at short notice. The group was also involved in provision of logistical support for special visits and events in the life of the ILL.

#### Services for external organisations

The group carried out maintenance of the building and the technical installations of EMBL outstation and of the temporary building of the ESRF. The major work in 1987 has included the transfer of the concrete prefabrication zone of the ILL and the studies for relocation of the buildings ILL9, ILL10, ILL14, ILL23, ILL24 and for modifications of the network of roads and drainage on the ILL site.

# INSTRUMENT GROUP FUNDAMENTAL AND NUCLEAR PHYSICS

- PN1 : Fission product separator (LOHENGRIN) on beam tube H8 (H.R. Faust, J.P. Bocquet, R. Brissot, I. Gartshore).
- PN2 : Beta spectrometer (BILL) on the vertical beam tube V3 (B. Krusche, K. Schreckenbach, 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, S. Dewey, R. Oliver).
- PN4 : Ge(Li) pair spectrometer on beam tube H7 (S. Robinson, R. Oliver)
- SN5 : Ultra-cold (UCN) and very cold (VCN) neutron source with 2 beam positions on the inclined beam tube IH3 (P. Ageron, W. Mampe, A. Beynet)
- SN6 : On-line mass separator for thermally ionised fission products (OSTIS) on neutron guide H23L (U. Stöhlker, W. Lippert, V. Rabbel, T. Manning)
- SN7 : Cold polarized neutron beam at end position of guide H14 (K. Schreckenbach, D. Dubbers)
- PN8 : Fission product coincidence spectrometer (Cosi Fan Tutte), (P. Geltenbort, T. Manning)
- H17 : Cold neutron guide with liquid helium UCN source (P. Ageron, I.A. Kilvington)
- H18 : Cold neutron guide (W. Mampe)
- H22 : Thermal neutron guide: neutron induced particle emission (H22D), (J.P. Bocquet; prompt gamma activation analysis (H22E), (S. Robinson, R. Oliver);  $\gamma$ - $\gamma$  angular correlations (H22 F), (S. Robinson, R. Oliver)
- TGV : Vertical VCN guide in cold source connected to Steyerl turbine producing UCN (Niveau D), (P. Ageron, W. Drexel, W. Mampe, A. Beynet, A. Steyerl).

## INSTRUMENT IMPROVEMENTS AND DEVELOPMENTS

### Fission research

Fission experiments were performed on three machines: PN1, PN8 and SN6.

#### PN1:

The effort on the instrumental side on PN1 was concentrated on the development of the large ionisation chamber which opens the possibility to include nuclear charge measurements of fission products via the E- $\Delta E$  method. It was found that a considerable part of the straggling of the energy loss in an ionisation chamber with a divided anode is due to the diffusion of the electron cloud. This leads to a cross talk of the ionisation signal on both parts of the anode. This diffusion was avoided by the introduction of a grounded grid in the

chamber, separating the gas volume of the E and  $\Delta E$  part electrically (see photograph). Further improvement has been made on a proper design of the entrance window of the chamber. Fig. 95 shows an example of the nuclear charge resolution of the chamber which uses 7 cm of the focal plane of Lohengrin. A comparison is given on Fig. 96 between the new method and results obtained with a passive absorber. Although the active method does not reach the nuclear charge resolution obtained with the passive absorber, it offers the possibility of discrimination against contaminating lines: As a consequence the sensitivity was enhanced sufficiently to allow the identification of fragments produced at rates of  $10^{-9}$  per fission (i.e. one fission product per day).

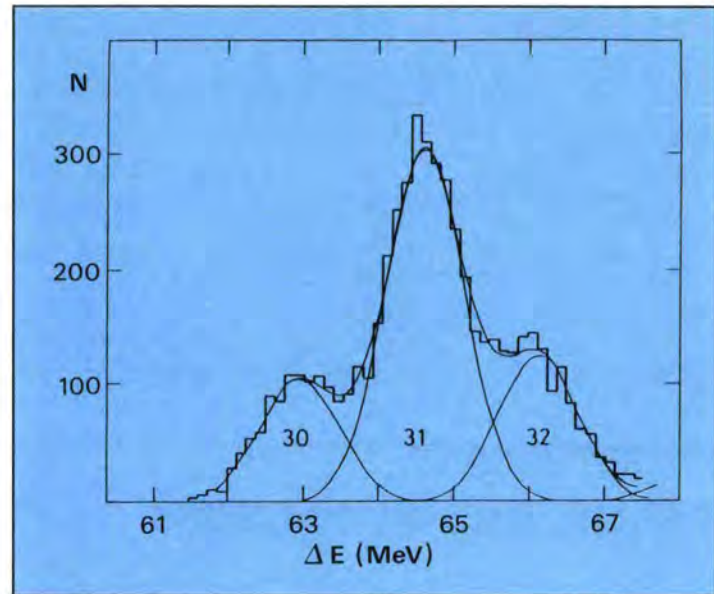
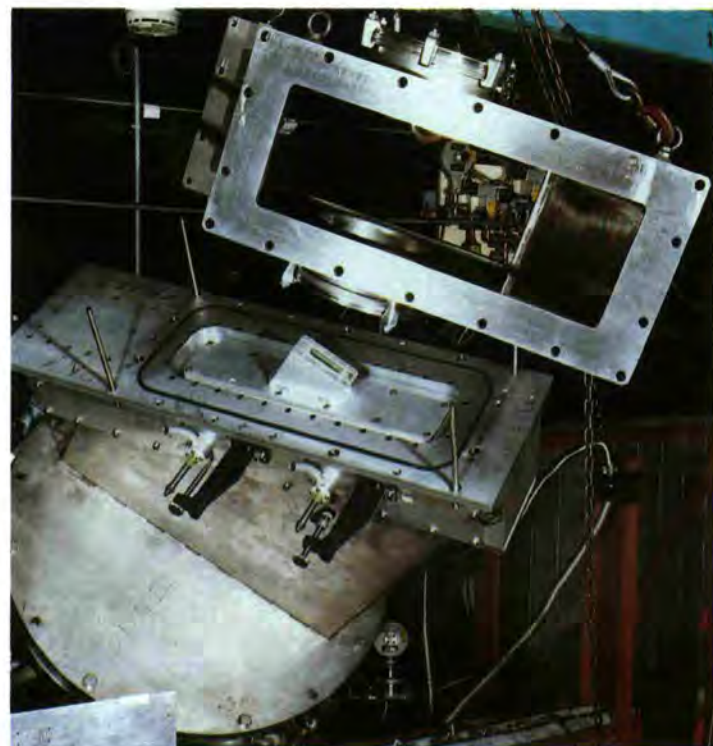


Figure 95: Z-resolution of the chamber



View of the ionization chamber for E $\Delta$ E measurements.

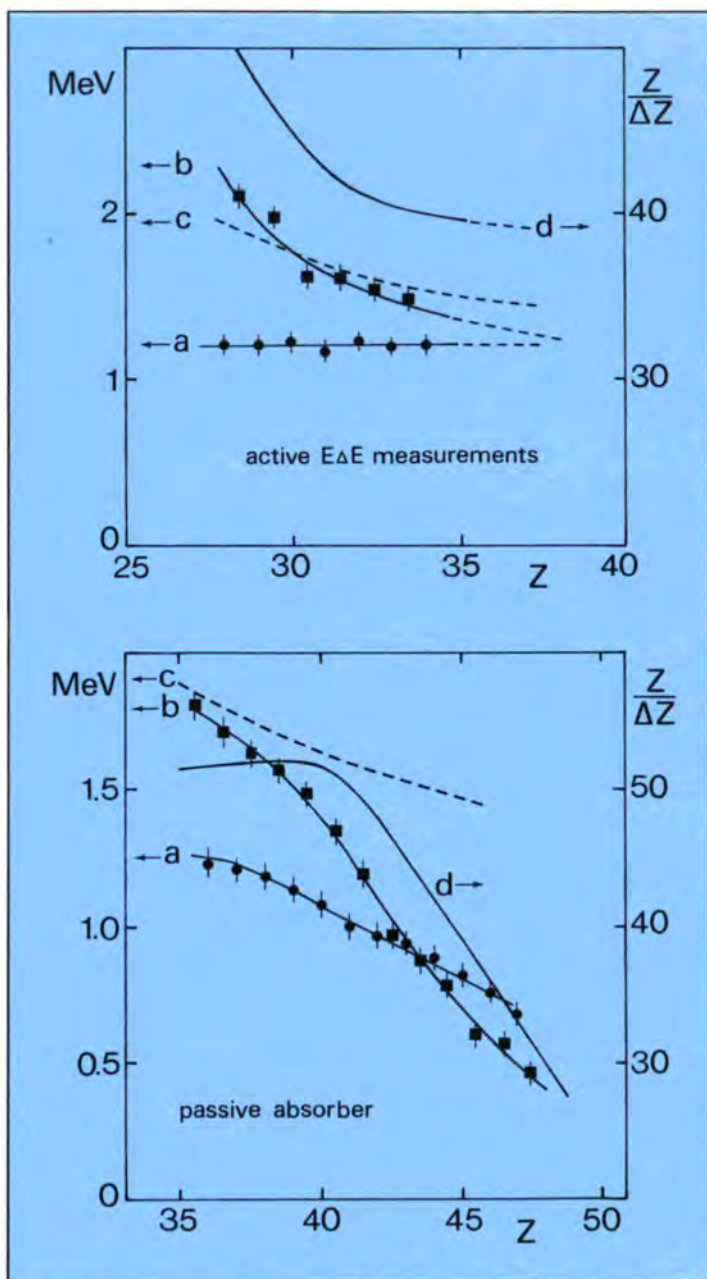


Figure 96: Comparison of the Z-resolution in passive and active measurements  
 (a) widths of residual energy peak  
 (b) distance between two neighbouring charges  
 (c) semi empirical estimate for (b)  
 (d) charge resolving power

#### PN8:

Cosi fan tutte operated in 1987 as scheduled in a fully satisfactory way. The instrument has also been used in a new mode: One of the two originally identical spectrometer arms had been shortened by the time of flight detector section (see Fig. 97). This way the axial ionisation chamber gets much closer to the target. As a consequence the efficiency for coincidence measurements following binary fission is increased by a factor of 10. This increase is necessary because the initial collinearity of the two fission fragments is partly lost by small angle scattering and neutron emission. The missing velocity information on one fission product can be obtained from symmetry considerations giving finally access to the not directly measurable fragment quantities before neutron evaporation. Both configurations will be available for neutron experiments.

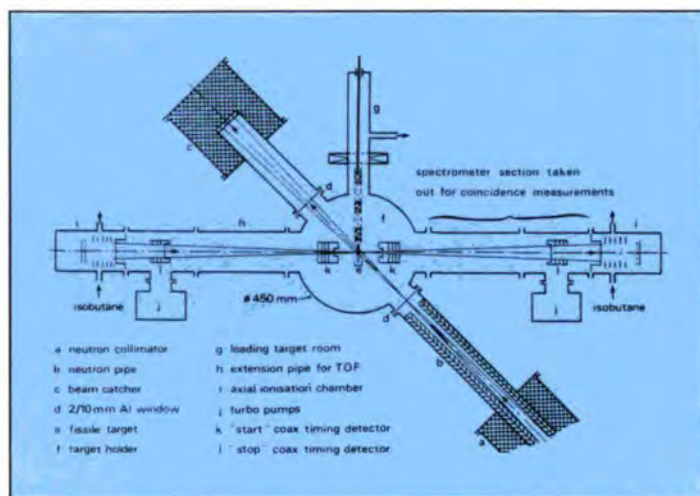


Figure 97: Schematic view of the coincidence fission fragment spectrometer "cosi fan tutte" (PN8) (symmetric set-up with two long arms)

#### SN6:

Several improvements have been carried out on Ostis: modernisation of the vacuum system, construction of a target test facility, redesign of the target holder leading to reduced diffusion times of the fission products in the source, installation of a new magnetic electron separator for more precise Q-beta measurements. A neutron trumpet for flux increase on the fission target has been nearly completed.

## Spectroscopy

#### PN2:

The high resolution conversion electron spectrometer BILL continued to run reliably during 1987 covering a wide range of experiments. Besides minor modifications (upgrade of the computer memory etc.) some efforts were made to optimize the use of the spectrometer as a high resolution positron source for scattering experiments (see also scientific part of College 3). The semiconductor detectors in these studies needed new diffusion pumps at the spectrometer section of the beam tube with almost complete suppression of retrodiffusion. In the latest version the in-pile source was a  $(90 \times 50 \times K 3) \text{ mm}^3$  sheet of natural titanium. In this sheet both positrons and electrons are produced following neutron capture reactions. For the appropriate direction of the magnetic fields only the positrons (resp. electrons) can pass through the spectrometer magnets where they are momentum analysed and focussed onto the focal plane. Fig. 98 shows the intensity of the electron and positron beam measured in the focal plane with a 32 wire detector as a function of the energy. The length of the segment of the focal plane viewed by this detector is approximately 64 mm corresponding to an energy dispersion of  $\approx 10 \text{ keV}$  at 1 MeV and  $\approx 2 \text{ keV}$  of 2 MeV. However, it should be emphasized that this dispersion does not pose a severe limitation to the energy resolution achievable in scattering experiments. In a typical set-up the spatial resolution of about 20 mm obtained by a multidetector arrangement permits to correct for the dispersion and thus the energy spread of the incident positrons is limited to  $\approx 5 \text{ keV}$  at 1 MeV even if a larger section of the focal plane is used. In Fig. 98 no values are given for the electron intensity between 1 MeV and 4 MeV. In this region the count rates are higher than 1 MHz and the multi-wire proportional counter saturates. Both spectra show a pronounced step-

like structure, the electrons around 6.5 MeV and the positrons around 5.5 MeV. This is caused by three strong primary transitions from the reaction  $^{48}\text{Ti}(n,\gamma)$  with energies of 6418 keV, 6556 keV and 6760 keV and intensities (photons per 100 captured neutrons in natural Ti) of 29 %, 5 % and 44 % respectively. The electron spectrum is governed by  $\gamma$  self-absorption and internal conversion whereas the positron spectrum is entirely due to external and internal pair production, which explains the  $\approx 1$  MeV shift of the edges. It should be noted that the positron spectrum is almost constant and at a maximum value between 1-3 MeV which is the region of special interest for the present studies of the Bhabba excitation function. This maximum intensity is comparable to those currently achieved at accelerators in the same type of experiments, but the Bill spectrometer provides high energy resolution, good geometrical stability of the beam spot and the opportunity of very easily tunable beam energy.

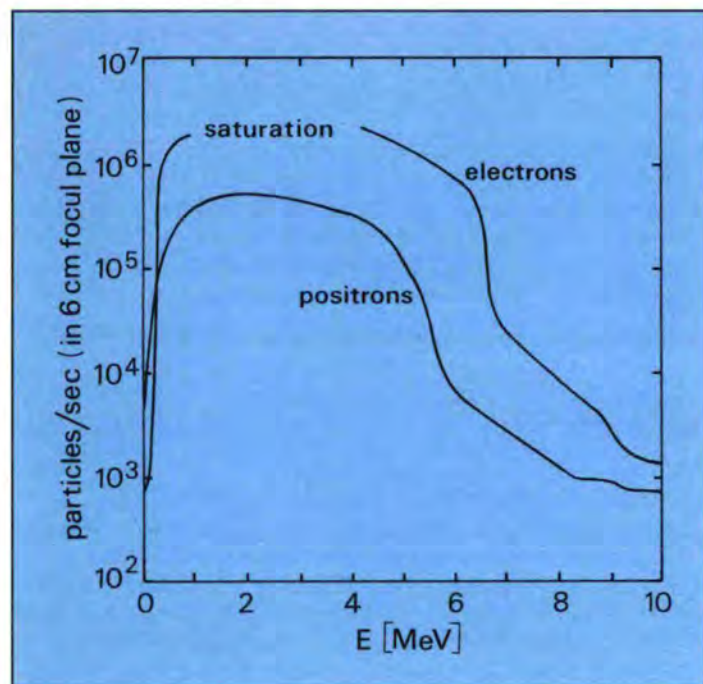


Figure 98: Electron and positron intensity, resp. measured in the focal plane with a 32 wire detector

It is probably not possible to use even thicker Ti plates for further increase of the beam intensity (heating problems), however other materials are under consideration.

### PN3 and PN4:

A major improvement in background reduction was achieved in replacing the  $5 \times 5$  cm<sup>2</sup> NaI - detector mounted at Gams 1 by a n-type HPGe - detector with a relative efficiency of 25 % and a resolution of 2.1 keV at 1.33 MeV. There are three different sources of background:

- i) ambient background, which can be reduced by heavy shielding and by having the reflected intensity in a small energy window due to the good resolution of the detector.
- ii) background from  $\gamma$ -rays from the core but also from the target which are scattered at the source and fulfill the Bragg condition at the crystal. This background reaches its maximum at an energy of around 180 keV. It can be reduced partly in using a Ge-detector with good resolution since many of these  $\gamma$ -rays scatter off the graphite holder and are therefore out of focus. The width of this radiation coming from a 0.5 cm wide

source (the maximal dimension of the graphite holder) corresponds to  $\approx 150$  arcsec resulting in an energy width after the crystal of  $\Delta E \approx 18$  keV at a Bragg angle corresponding to 200 keV  $\gamma$ -ray energy.

iii) background created by direct radiation which is scattered in the soller collimator and reaches the detector. This background is most important for high energies (small Bragg angles). As for i) the gain in background suppression is given by the ratio of the resolution of the detecting devices. A further advantage is the reduction of cross talk between the different orders. At low energies the spacing between different orders is such that they cannot be completely resolved with a NaI-detector, so that each line in a given order will influence the neighbouring orders. Moreover the amount of Compton events in the detector recorded in lower orders is reduced considerably using a Ge-detector.

First tests have been performed using a new crystal bender, which allows to adjust the radius of curvature over a wide range. This will permit to perform measurements making full use of the second target position in a reduced neutron flux discussed in the 1985 report. The distance from the GAMS2/3 spectrometers to this position is 23.5 m instead of the 24m to the high flux position. The crystal to be used in this bender is of triangular shape, clamped on its base and pushed at its top, to give the cylindrical bending required by the Du Mond - geometry. An almost perfect Si crystal, cut to provide the (2n,2n,0) reflections, and having a thickness of 1.7 mm is used in the present bending device. The motion of the top perpendicular to the base is 100  $\mu$  for a radius of 24m and 102 $\mu$  for 23.5 m. This motion is accomplished by the use of a linear translation stage, allowing adjustments with a precision better than 0.01  $\mu$ . First measurements using a  $^{197}\text{Au}$  target (thickness 15  $\mu$ ) resulted in an angular resolution of  $\approx 0.2$  arcsec using a window of  $1.5 \approx 1.3$  cm<sup>2</sup> in front of the crystal. Preliminary data show that the reflectivity remains constant at least up to 750 keV in second order.

The resolution using the classical quartz crystal was also improved as compared to previously: The limitation in resolution obtained at the GAMS instruments is given by the quality of the targets and the reproducibility of the adjustment procedure. The source material was enclosed in  $40 \times 4$  mm<sup>2</sup> aluminium lamina and pressed to yield sources of about 30  $\mu$  thickness and a flatness better than 10  $\mu$ . These lamina are enclosed into graphite holders being machined to a flatness of better than 10 $\mu$ . The design of the graphite holder is made such that a maximum of stability is reached in the source holder tube. The resolution of 0.4 arcsec shown in Fig. 99 was maintained during several days using a crystal surface of  $\sim 8$  cm<sup>2</sup> and a standard  $4 \times 40$  mm<sup>2</sup> source containing  $\sim 20$  mg of  $^{187}\text{Os}$  metal. Since this resolution corresponds to an effective source thickness of only 45  $\mu$  one has to conclude that the resolution of the GAMS2/3 spectrometers still is dominated by source effects and might be improved further.

With the two axis flat crystal spectrometer, GAMS4, a resolution of  $\Delta E/E \sim 10^{-5}$  can currently be achieved for 1.5 MeV  $\gamma$ -rays measured in 2nd order of reflection using a nearly perfect Si(220) crystal. Amongst others this excellent resolution can be used to measure Doppler broadened  $\gamma$ -rays (and hence determine lifetimes of excited states). The analysis of these lineshapes requires the precise knowledge of the instrumental response function which depends strongly on the energy. Here we want to comment on the impressive feature of self-calibration used for this purpose in the double flat crystal method: This method involves the rocking of the second crystal so that its orientation is  $+/- \Theta_{\text{Bragg}}$  with respect

to the beam diffracted at  $+\Theta_{\text{Bragg}}$  at the first crystal ( $\sin \Theta_{\text{Bragg}} = n\lambda/2d$ ). An important characteristic is that in the  $+\Theta_{\text{Bragg}}$  mode the two crystal corresponds to a non dispersive mode if the  $\gamma$ -ray is reflected by both crystals in the same reflection order  $n_1 = n_2$  and hence the measurement reproduces the response function of the instrument at  $\Theta_{\text{Bragg}}$ . At  $-\Theta_{\text{Bragg}}$  the instrument operates in a dispersive mode and such measures the energy distribution of the observed  $\gamma$ -ray (besides a vertical divergence correction due to the finite size of target and crystal but which: a) is small ( $\leq 10^{-6}$ ) and b) can be described in closed form). Therefore, by measuring each  $\gamma$ -ray at  $+\Theta_{\text{B}}$  (Fig. 100, left side) and  $-\Theta_{\text{B}}$  (Fig. 100, right side) one obtains a) the wavelength  $\lambda$  from the angle  $2\Theta_{\text{B}}$  and b) the energy distribution measured at  $-\Theta_{\text{B}}$ .

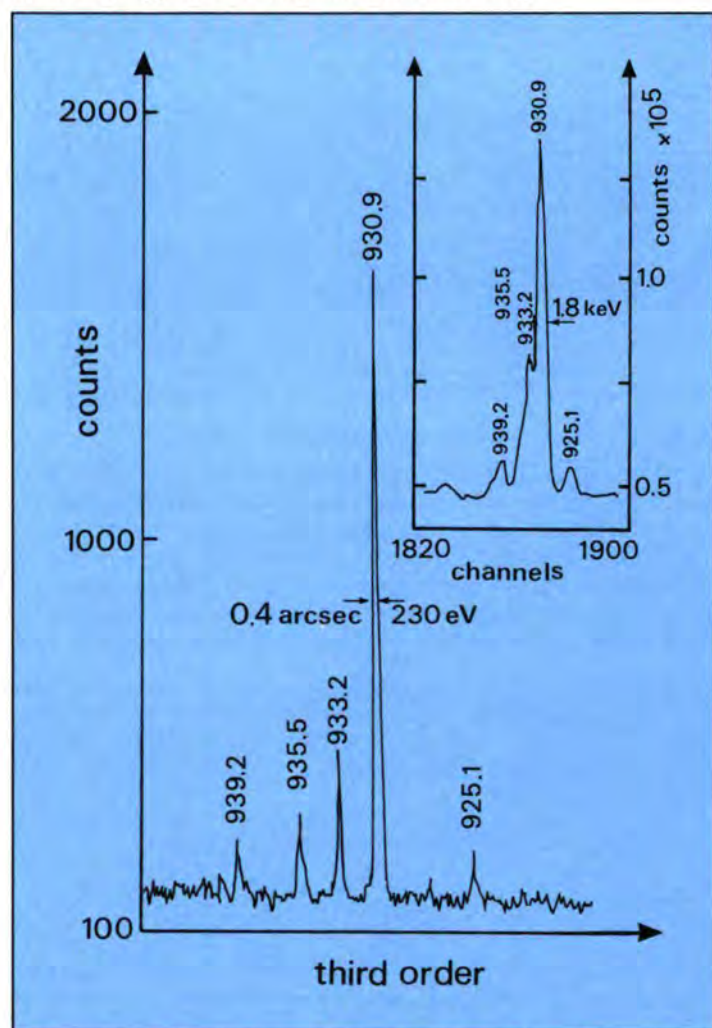


Figure 99: Resolution obtained with GAMS2 compared with the resolution of a HPGc detector measuring the same target

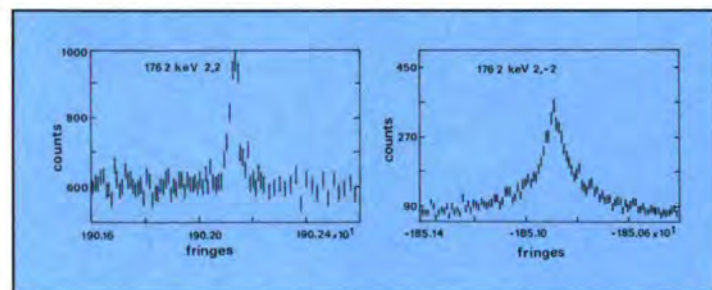


Figure 100: Intensity profiles of a 1762 keV transition in  $^{49}\text{Ti}$  measured in the dispersive (2, -2) and non-dispersive (2, 2) mode of the GAMS4 spectrometer. The width of the non-dispersive line amounts to approximately 30eV.

## FUNDAMENTAL PHYSICS

The long term experiments in fundamental physics have been continued throughout 1987 on ultra-cold, very cold and thermal beams and new instrumental developments have been started. The trend to innovations persists and a steadily increasing interest of external groups, which realize the outstanding possibilities of the high flux reactor for research in fundamental physics with neutrons, can be observed.

### SN5:

The remaining two beams of the first UCN-VCN source were used for preparing experiments since all beam ports of the new, much stronger TGV source had been occupied: the VCN beam served a series of different reflectivity experiments including control measurements of the second cold source guides. The UCN beam was used for development work on the electric dipole moment experiment such as: test of various UCN storage bottle materials, new UCN polarizers, study of bottle filling and emptying behaviour, etc. A new high resolution energy selector has been designed and constructed by the Sussex group with the aim to complete the EDM work and possibly the neutron lifetime studies by additional information on the energy distribution of the remaining ultra-cold neutrons.

### Experiments on the TGV-turbine source:

The long term experiments using UCN and VCN took data in 1987 as described in more detail in the scientific section of the report. In parallel the following instrumental improvements have been performed:

The neutron storage ring NESTOR installed on the VCN beam was complemented by an additional detector allowing for automatic axial scanning of the neutron beam profile. The reproducibility of the diaphragm control, necessary for precise beam shaping in the neutron half life experiment has been improved as well by the Bonn group.

A new HV stack (max 300 kV) had been built at RAL and connected to the EDM experiment by a longer cable able to withstand 300 kV. A new HV feed through into the vacuum is expected to eliminate a small HV insulation oil leak which was probably the cause of a slow degradation of the UCN bottle storage qualities and the simultaneous increase of the leakage current. The HV characteristics have thus been improved.

The improved neutron microscope version had been replaced by an UCN diffractometer using the dispersion of the gravitational field for neutron energy determination (see scientific + special beam section).

The neutron lifetime experiment using storage of UCN in material bottles had been temporarily moved from the test beam position to the one foreseen for the gravity spectrometer NESSIE from Garching. The gain in intensity was four fold but the spectrum is harder. The vacuum was improved to  $2 \times 10^{-7}$  bar and the bottle temperature stabilized to  $\pm 0.1^\circ\text{C}$ . A new UCN detector is under study.

### SN7:

The end position SN7 of the cold polarized neutron beam H14 was again very demanded for fundamental physics experiments through 1987 (see College 3). A thermo-isolated cabin (area  $6 \times 5 \text{ m}^2$ , height 4.5 m) was built around the experimental area and improved significantly the instrumental stability and the working conditions. The following instrumental developments are briefly mentioned:

area and improved significantly the instrumental stability and the working conditions. The following instrumental developments are briefly mentioned:

For neutron lifetime measurements with pulsed neutron beams two different neutron-beam techniques were developed. In the first method the beam was reflected under  $90^\circ$  by a graphite or  $\text{RbC}_8$  crystal, yielding monochromatic neutrons ( $\sim 2\%$  resolution) of  $\lambda = 4.7 \text{ \AA}$  and  $8 \text{ \AA}$ , respectively. The beam was chopped in front of the monochromator by a drum of  $.50 \text{ cm}$  diameter rotating with  $\sim 3400 \text{ rpm}$  (Fig. 101). This type of a double chopper yields symmetric pulse shapes, reduces the background in the secondary beam and suppress higher order Bragg reflections of the monochromator due to its wavelength resolution of  $\sim 40\%$ . In fact the background in the drift-chamber was not significantly increased by the presence of the main neutron beam. The resulting neutron pulses were about  $20 \text{ cm}$  long and contained  $2000$  neutrons per pulse in a beam cross section of  $15 \times 25 \text{ mm}^2$ . In a second arrangement a single chopper was placed at  $340 \text{ cm}$  in front of the chopper drum and the detector was in line behind the drum. This three synchronised chopper device yielded  $\sim 10000$  neutrons per pulse of  $30 \text{ cm}$  length and  $15 \sim 25 \text{ mm}$  cross section. The resolution was  $\Delta\lambda/\lambda = 8\%$  (full width) at  $7 \text{ \AA}$ . The background conditions were not as favourable as with the  $90^\circ$  deflected beam, but improvements are possible.

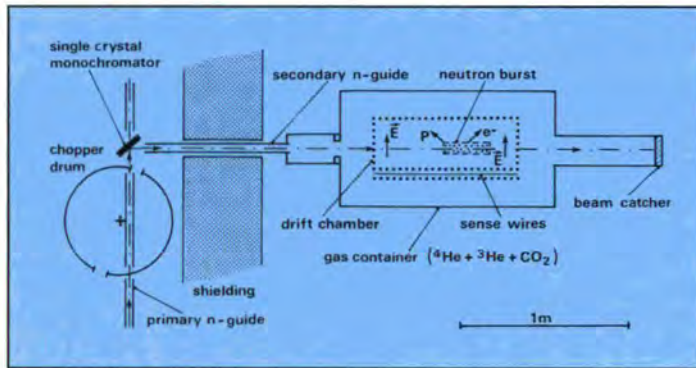


Figure 101: Experimental set-up of the neutron life-time experiment on a chopped monochromatized neutron beam with a He filled drift chamber.

### “Zero-Field Neutron Spin-Echo”:

A novel “zero-Field Neutron Spin-Echo” principle was tested with the apparatus sketched in Fig. 102. The neutron’s flight path within the instrument has zero magnetic field, and spin-echo response is induced by appropriate phase-locked magnetic resonance spin flips. The method has promising features and may allow for neutron spin-echo spectroscopy with very long base-lengths or under  $4\pi$ -detection (see Fig. 103).

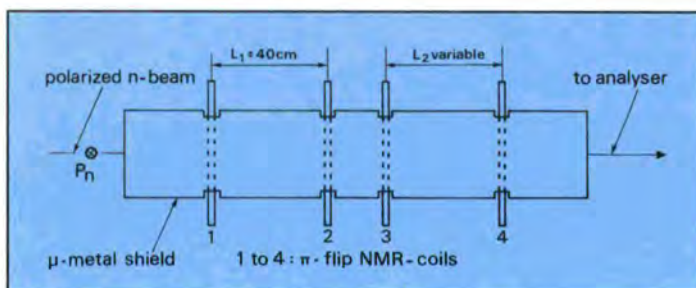


Figure 102: Layout of the small zero-Field Neutron Spin-Echo prototype instrument

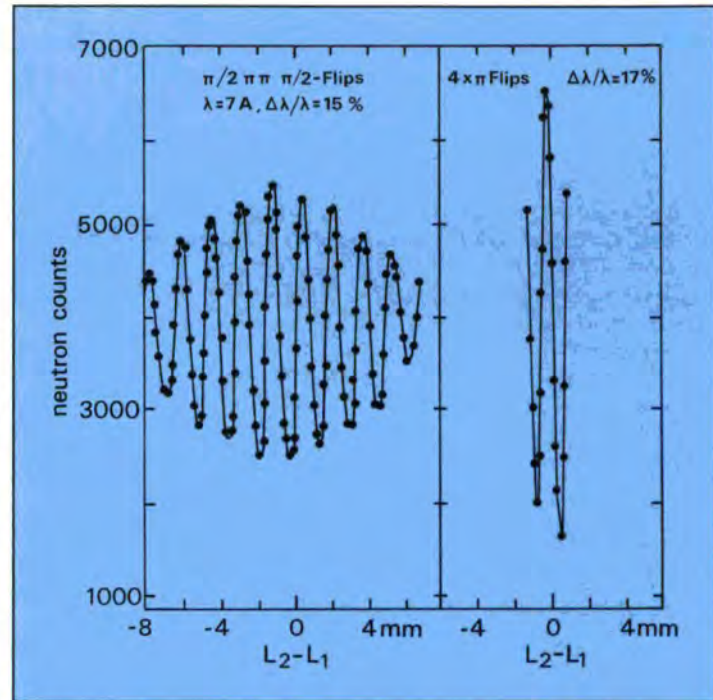


Figure 103: Neutron Spin-Echo signals obtained with the apparatus of Fig. 102. Two different flip-sequences have been tested. The resolution is that of a  $2 \times 0.4 \text{ m}$ ,  $200 \text{ gauss}$  conventional NSE spectrometer.

### A Broad-Band Neutron Spin-Flipper:

Some new spin-flipping devices based on a static magnetic field have been developed which have an almost  $100\%$  flipping efficiency over a broad range of neutron velocities. These devices are applicable both for  $\pi/2$  and  $\pi$ -spin echo flips. Fig. 104 shows the response of such a broadband  $\pi$ -flipper. When placed between two super-mirror polarizers, such flippers can also be used as velocity filters for polarised neutrons, see Fig. 105.

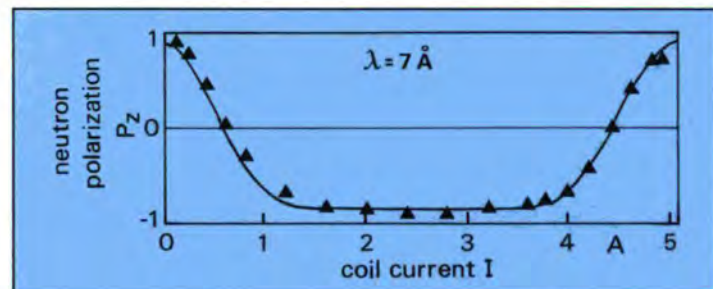


Figure 104: Polarization transmission characteristic of a broadband static field  $\pi$ -flipper. The response is the same when not the coil current but the neutron wavelength is varied.

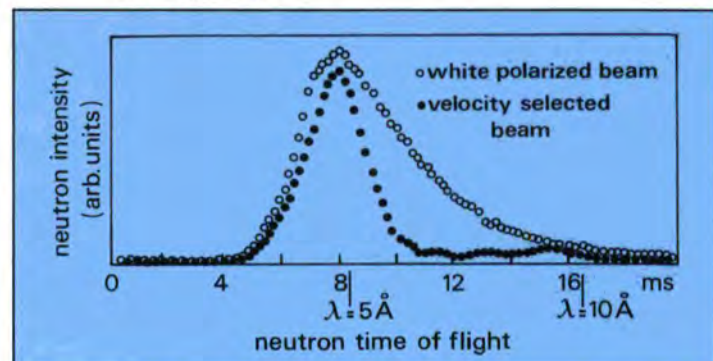


Figure 105: Broad-band  $\pi$ -Flipper used as a velocity filter for polarized neutrons.

The liquid Helium UCN source has been reinstalled on H17, a He-4 purifier has been built and the low temperature UCN exist system is under preparation for installation. No modification had been undertaken on H18 whereas the final instrument (S50) for the determination of  $h/m_n$  has been installed by the PTB-group in the guide hall.

Co-ordinator: W. Mampe

## **INSTRUMENT GROUP THREE-AXIS SPECTROMETERS**

- IN1 : 3-axis and beryllium-filter spectrometer on the hot source beam-tube H8  
(B. Dorner, H.J. Lauter, J.P. Varini).
- IN3 : 3-axis spectrometer on the thermal guide-tube H24  
(H.J. Lauter, L. Cussen, V. Frank, R. Arthaud).
- IN8 : 3-axis spectrometer on the thermal beam-tube H10  
(R. Currat, M. Alba, C. Vettier, A. Brochier).
- IN12: 3-axis spectrometer on the cold guide-tube H142  
(W.G. Stirling, S.M. Hayden, H. Godfrin, D. Puschner).
- IN14: 3-axis spectrometer on the cold guide-tube H53 on the second cold source.  
(W.G. Stirling, R. Currat, W. Kaiser, A. Brochier).
- IN20: 3-axis spectrometer for neutron polarisation analysis on the thermal beam-tube H13.  
(P. Frings, C. Vettier, J.L. Martinez, P.Flores).

### **GENERAL**

As already mentioned in last year's report, there has been great concern about the staffing in the group. Four scientists left our group last year and this situation has worsened seriously with the recent departure of W.G. Stirling to the University of Keele, UK, especially when new projects such as IN14 require the presence of dynamic and experienced scientists. The manpower shortage has been only partly compensated by the arrival of M. Alba, J.L. Martinez and S.M. Hayden. Nevertheless progress towards more reliable and user-friendly instruments has been made. In particular, a great deal of effort has been spent for improving the data processing as well as the efficiency of software on instruments by P. Frings.

### **INSTRUMENT IMPROVEMENTS**

#### **IN1: Hot source 3-axis spectrometer (H8).**

The instrument was operated reliably over the scheduled period, which has been slightly extended at the expense of the Be-filter instrument. The Si filter has definitively been removed because it has proved to be useful in the thermal neutron energy range but not in the case of hot neutrons. Furthermore, a Si filter seems to be more appropriate for a radial beam tube than a tangential tube like H8. Peripherals for data analysis available at the instrument have also been improved.

#### **IN1: Be-filter (H8).**

The instrument ran without problems. The Be-filter is used in combination with the Cu(200) and Cu(220) monochromator crystals in order to match the 5 meV resolution of the filter itself. The Be-graphite combination filter with a resolution of

less than 2 meV usually requires the Cu(331) monochromator crystal. A comparison between this spectrometer and the TFXA-instrument at ISIS is in progress. During this comparison a new graphite filter with a higher transmission will be tested.

### **IN3: 3-axis spectrometer on the thermal guide (H24).**

The instrument has been equipped with a closed-loop control in particular for the D.C. Tanzboden motors. The adjustment of the PID parameters for a correct positioning of the instrument arms driven by these motors is now almost completed.

The double-faced monochromator (PG(002) and Cu(111)) has been installed. Both monochromators have adjustable vertical curvature which can be varied during a scan. The monochromators can be changed automatically. The horizontally curved analyser is often used in semi-powder studies of surfaces or in studies where dispersionless excitations are present in order to gain a factor of about 4 in signal; furthermore an option has been installed in the instrument program which can set the sample orientation angle to give the focusing condition for a plate shaped sample. The analyser crystal support is currently being reconstructed to give better stability to the nearly free standing crystals. A new bending mechanism is envisaged to make the device more compact.

A change in position of the instrument to the end of the H25 guide would provide much more flexibility, dynamical range and incident flux.

### **IN8: 3-axis spectrometer on the thermal beam (H10).**

The instrument IN8 has been fully operational throughout the year. The renewal of the primary beam geometry has been completed by installing a new 15x15 cm<sup>2</sup> Ge(111) monochromator crystal with variable vertical curvature and a new mechanical drive for optional in-pile Soller collimators (15' 30' and 50'). Tests to quantify the high-order contamination at various wavelengths have been performed. At  $\kappa_1 = 2.662 \text{ \AA}^{-1}$ , actual flux values for the nominal  $\kappa_1$  are  $6.4 \cdot 10^7 \text{ n/cm}^2/\text{s}$ ,  $3.6 \cdot 10^7 \text{ n/cm}^2/\text{s}$ ,  $1.9 \cdot 10^7 \text{ n/cm}^2/\text{s}$  for PG(002), Ge(111) and Cu(111) crystals respectively, with 40' collimation before the sample and without PG filter. Using the IN8 PG filter would reduce these values by 22%.

Two remote control devices, namely a pyrolytic graphite filter and a plexiglass handler, have been developed to improve the flexibility of the instrument during sample alignment procedures. These devices will be soon fully computer-controlled. The upgrade of the computer memory up to 4 MB and the more user-friendly programs have greatly improved the efficiency of the on-line reduction of raw data. Furthermore it is worth noticing that better acoustic insulation from neighbouring noise sources has led to a real improvement in the noise level.

Further developments will include feasibility tests of horizontally focusing analyser crystals.

### **IN12: 3-axis spectrometer on the cold neutron guide (H42).**

IN12 continued to run reliably although some mechanical problems developed at the end of the scheduled period. These were

found to be due to wear on the brakes which fix the translation of the sample table and have now been corrected. At the end of the year we began the work of replacing the original electronics by a closed-loop/DC motor system. The SOLAR 16-40 computer is also to be changed for a PDP 11-73. This will bring a number of new parameters under direct control of the computer including the guide fields and flippers used in polarised work and the sample goniometer. IN12 will operate under the same spectrometer control program (TAS) as the other instruments in the triple axis group.

### **IN14: 3-axis spectrometer on the horizontal cold source (H53 guide).**

During the course of the year the design of the instrument was completed and the construction stage has now started.

The monochromator protection was assembled and tested mechanically at the construction site (see photograph) and is now ready for final installation (Dec.87 - Jan.88).



*Monochromator drum for IN14, the new 3-axis spectrometer on the second cold source.*

Concerning the monochromator axis, the sample table and the secondary spectrometer, most mechanical parts have been supplied and assembly work will soon begin in the Hall d'Essais. The analyser and detector protections will be constructed by external contractors (delivery date: Feb.88). The instrument will be equipped with vertically focusing PG(002) and Si(311) monochromator crystals, a magnetic bender for polarised neutron work, graphite and Heusler crystals as analysers, a cooled Be-filter, a conventional PG-filter ( $\lambda = 2.35 \text{ \AA}$ ), and a continuous wavelength (D17-type) PG-filter ( $2.35 \text{ \AA} < \lambda < 4.05 \text{ \AA}$ ) for the suppression of higher order neutrons. Some of these items have already been delivered, others are due for delivery in early 1988. The control electronics includes Moore-Reed shaft-encoders, closed-loop motor control modules, a CAMAC interface and a PDP 11/73. All major hardware parts have been supplied.

The first neutron tests are planned for the 3rd reactor cycle (May 88).

## IN20: 3-axis spectrometer with polarisation analysis on thermal beam (H13).

1987 was the first complete year that IN20 was in full, routine operation. As was expected for a relatively new instrument, several problems have surfaced during this period.

One of the major problems was the lack of mechanical reproducibility of the primary spectrometer. This problem was finally discovered to be due to an underdimensioned attachment of the sample table to the monochromator drum. After modification and a complete optical realignment, the machine is now working satisfactorily concerning this aspect. An optical check of the monochromator crystal turret is now possible. On the analyser side, dismounting and mounting analyser crystals may introduce angular offsets. This mechanical problem will be repaired before the end of the year. On the polarised neutron part two major items were or will be improved. The first, already implemented, modification concerns the flipper after the sample: by moving it to a position much further away from the sample it became about one order of magnitude less sensitive to the magnetic sample environment, which guarantees a constant flipping ratio over the whole range of accessible scattering angles. Another limitation was due to the Helmholtz coils around the sample which limited the continuously accessible scattering angle to parts of 30 degrees. A newly developed coil-system will overcome this difficulties and offer free access over 110 degrees in scattering angle.

Last but not least the speed and reliability were greatly improved by the installation of a new PDP 11/73 and several modifications to the software.

Future developments will probably concentrate on an extension of the polarised neutron range to higher energies and a calibration on an absolute scale of the sensitivity of the instrument. As a first step we measured carefully the amount of higher order contamination counted by the monitor as a function of the incoming energy for the different monochromators. Actual flux values without PG filter but corrected for high order contamination at  $k_1 = 2.662 \text{ \AA}^{-1}$  are  $5.5 \cdot 10^7 \text{ n/cm}^2/\text{s}$  for a PG(002) crystal and  $1.2 \cdot 10^7 \text{ n/cm}^2/\text{s}$  for a Heusler(111) crystal with  $40^\circ$  collimation in front of the sample and no PG filter.

Coordinator: C. Vettier

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

- IN4 : Time of flight spectrometer on thermal tube H12 (A. Murani, H. Mutka, M. Belmonte).
- IN5 : Multi-chopper spectrometer on cold guide H16 (G. Kearley, H. Blank, S. Jenkins).
- IN6 : Focussing TOF spectrometer on cold guide H15 (A.J. Dianoux, B. Frick, Y. Blanc).
- IN10 : Backscattering spectrometer on cold guide H15 (A. Magerl, S. Mahling-Ennaoui, P. Joubert).
- IN10C : (project) New backscattering spectrometer (A. Magerl, J.L. Coquin, Y. Blanc).
- IN11 : Spin-echo spectrometer on cold guide H142 (B. Sarkissian, B. Farago, J. Bauchat).
- IN11B : (project) Multidetector spin-echo spectrometer on cold guide H142. (B. Sarkissian, J.L. Coquin, J. Bauchat).
- IN13 : Backscattering spectrometer for short wavelengths on thermal guide H24. (W. Petry, J. Eckert, J.F. Barthélémy).
- IN15 : (project) Spin-echo spectrometer for long wavelengths (F. Mezei, D. Richter, C. Lartigue, F. Douchin).
- D7 : Diffuse scattering instrument with polarization analysis on cold guide H15. (O. Schärpf, W. Just, R. Rebesco).
- D11 : Small angle scattering diffractometer on cold guide H15. (P. Timmins, A. Wright, R. May, R. Baker).
- D17 : Low Q, high resolution diffractometer on cold guide H16 (A. Rennie, J. Torbet, R. May, M. Cruz).
- D22 : Project of small angle scattering diffractometer on the Horizontal Cold Source (R. May, M. Thomas).  
Group Engineer: F. Douchin.

## IN4: TOF spectrometer on thermal beam H12

Operation of the spectrometer in the single monochromator configuration has been very satisfactory during the year. It is used increasingly for experiments which can be performed with neutrons of incident energies 17, 68 or 115 meV available with the graphite (002), (004) and copper (220) monochromators. For the 68 meV incident energy a flux gain at the sample position of roughly an order of magnitude (10 times) is noted compared with the copper double monochromator configuration at the same energy, but with slightly poorer energy resolution in the former case. The gain factor is over 3 with respect to the graphite double monochromator operation at  $E_i = 50 \text{ meV}$ , the energy resolution being the same (3 meV fwhm) in both cases. (i.e. a better  $\Delta E/E$  for the single monochromator arrangement). Incorporation of a collimator within the sample chamber has helped to reduce significantly

the background scattering due to the walls of the cryostat (or other sample environment device).

Decision regarding further improvements proposed several years ago including a shortened flight path with a multi-row detector arrangement, variable curvature four-face monochromator system, a new type of small angle detector, etc. is still awaited. If carried out these changes will significantly improve the spectrometer performance.

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

Fully automatic control of the chopper system, time-of-flight electronics and temperature controller now enable the full flexibility of this spectrometer to be exploited. Since the sample environment is usually a helium cryostat, the automation has been extended to include control of the cold valve and liquid helium transfers. A high-speed liquid nitrogen cryo-oven is now also available (77-700 K).

The count-rate for most experiments has been increased considerably by the installation of 120 new detector tubes (IN6 type) and we expect a similar gain when the final detector-configuration (a further 120 detector-tubes) is achieved. We expect to achieve much better detector reliability now that there is no longer any need to move the detectors between experiments. This event was marked by a new coat of paint.

Preliminary results from a test using the "spare" D11 multidetector are encouraging and it is clear that there is considerable scope for using IN5 for small-angle inelastic scattering experiments. There are however, clear limitations due to the range of incident energies available from the guide, and the restricted secondary flight-path. More extensive investigation of this application is scheduled for the coming year.

### **IN6: TOF spectrometer on cold guide H15**

IN6 has worked very efficiently during 1987 and its performance has permitted 42 experiments to be performed during the 5 reactor cycles. The heavy demand for the instrument is leading to shorter time allocations on the average. Nevertheless a reasonable time (2 weeks) was given for the study of excitations in  $^3\text{He}$  -  $^4\text{He}$  mixtures. During this time, 3 concentrations at 3 pressures and various temperatures were investigated and the results are being compared with various theoretical predictions.

Some improvements have been realized on the ancillary equipment: a new heating and cooling loops permits an automatic control of the temperature between 100 and 600° K. An automatic cold valve has been installed on the cryostat and extensive tests have been carried out in order to optimize the temperature control programme. A new drawing for standard sample holders has been realized with a better protection of the O-ring and anodized threads. A much more powerful secondary pumping unit has been connected to the evacuated sample area.

During the long shutdown at the end of the year, a new computer (VAX work-station) has been tested on IN6. It will control the instrument at the beginning of 1988 and will permit to test the transfer of TOF data to the VAX central computer.

### **IN10: Cold neutron backscattering spectrometer**

The major improvement on IN10 in 1987 concerns the preparation and installation of novel data acquisition electronics based on the VME bus, the future ILL standard. A complete chain has become operational and on-site tests in manual mode (McIntosh controlled) have proven the superior performance and reliability of the system. Important software development has been necessary for appropriate system - and utility programs. Starting with the first cycle in 1988 IN10 will take data in parallel on the old CAMAC/PDP11 system and the new VME/ $\mu$ VAX set-up coupled to the new Ethernet cable in the neutron guide hall.

On the cryofurnace an additional 200 W heater has been added to the standard 50 W element. In addition direct cooling of the sample area with cold nitrogen gas allows very rapid temperature changes in the entire range from 2 K to 600 K. The instrument control electronics has been centralised by installing a new chopper control unit in the instrument cabin.

### **IN10C: New backscattering spectrometer at the 2nd cold source**

IN10C has changed from a planning phase into the construction phase and several orders on major instrument components such as the mounting and the mechanics of the first deflector, the shielding of the primary spectrometer or the focussing neutron guide have been ordered. First parts such as the graphite for the first deflector or the spherical monochromator support made out of carbon fiber based compound material have been received. Elements further down stream of the instrument like the monochromator drive system, the sample area or the polydetector are being designed.

Difficulties have been faced with the second deflector crystal mounted on a chopper wheel. Tests have revealed a surprisingly low mechanical stability of the graphite. Its properties are studied now in detail, and subsequently a new layout of this device with considerable less stress on the graphite crystal will be defined. The graphite for the mechanical tests is already from the ILL's own production.

A definite choice for the electronic/computer system will only be taken during 1988 awaiting the results on the new VME/ $\mu$ VAX configuration on IN10.

### **IN11: Spin-echo spectrometer on cold guide H142**

During the last year IN11 worked reliably without electrical or other failure.

The computer was equipped with a streamer for backup purposes. The experimental data are transferred to the VAX central computer and archived in the standard way.

A new transmission-polarizer was designed and ordered. It will be tested and installed by the end of next year.

### IN11B: Multidetector spin-echo spectrometer on cold guide H142

After extensive testing of the small angle multidetector extension of IN11 the results show that it did not reveal the expected gain. There were essentially two reasons for that:

The first is that the solid angle covered by the detectors was smaller than expected (loss of space due to the analyser-coil winding, big sample to detector distance).

The second is that the inhomogeneities of the big magnet proved to be too important to approach the performance of IN11.

Small angle studies need quite generally the best resolution of IN11. For that reason a team was formed to study the possible reorientation of the project. As the small angle ultra-high resolution NSE with multidetector (IN15) is under construction, the most likely reorientation will be the higher Q, high flux multidetector extension with a possible compromise concerning the resolution.

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

In September 1987 a new and more powerful instrument computer of a PDP 11/73 type with the time sharing system TSX has been installed. On that occasion all basic routines and the acquisition programme have been rewritten. As a consequence the users' access to IN13 has been very much simplified and a couple of commands, similar to those used on IN5, are sufficient to control the spectrometer. To alleviate the load of the computer, neutrons are counted by external scalars. Only after a couple of seconds those counts are transferred to the computer memory and stored in an adequate temperature or energy channel.



A new furnace for IN13.

In 1987 IN13 has been extensively used for studies of the dynamics of the glass transition profiling from the  $\mu\text{eV}$  resolution and the large accessible  $Q (< 5.5 \text{ \AA}^{-1})$  range. For the first time an experiment on coherent quasielastic scattering has been performed. Oriented samples of  $\text{C}_{20}\text{F}_{42}$ , the basic monomer of Teflon, exhibit a strong diffuse (Zwischenreflex) scattering around  $Q = 2.3 \text{ \AA}^{-1}$  and  $Q = 4.5 \text{ \AA}^{-1}$ . The IN13 measurements confirmed, that this coherent diffuse scattering goes along with a temperature dependent relaxation process, the frequency of which coincides with the window of IN13. On that occasion it should be noted that IN13 has a particular set of analyzer crystals with slightly relaxed resolution, but with a twofold increase in intensity which can be used for this kind of coherent critical scattering experiments.

### IN15: Neutron spin-echo spectrometer for long wavelengths at the second cold source.

Considerable progress has been made in the definition and design of the instrument.

The beam splitter (TiNi supermirrors) separating the H511 (IN15) and H512 (D22) guide has been mounted in October.

The prepolarizing guide, 16 meters long, between the beam splitter and the velocity selector is being constructed: the glass has been coated at HMI-Berlin by sputtering one GdTi antireflecting layer and one  $\text{Fe}_{40}\text{Co}_{60}$  layer; air-cooled coils are being wound on the housing; the power supply will be chosen to provide a field of 100G but with the possibility of giving 200G for one second to fully magnetize the FeCo layer (values determined after neutron tests).

The mechanics is rather advanced. The housing of the velocity selector and the first three choppers to be under vacuum is designed. For the spin-echo part of the spectrometer, the two beams in composite fibers and two rotation modules are ready. A complete assembly of this part will take place in early 1988.

For the  $\text{He}^3$  multidetector ( $32 \times 32$  channels), the usual glass plate of the first cathode has been replaced by a grid of thin BeCu wires (100 mm and steps of 1 mm). A low He pressure allows for a very thin Al-window (1 atm  $\text{He}^3$  + 0.3 atm.  $\text{CH}_4$ ). Neutron tests will be performed from January 1988.

Great care is given to the conception of the precession coils since inhomogeneities of the order of  $10^{-5} - 10^{-6}$  can only be achieved if the cylindrical symmetry of the coils is perfect over their whole length. A final decision should be taken before the end of 1987. Calculations of the field integrals, accounting for a large beam diameter at the sample when using the focalisation, have shown that there is an optimal position of the Fresnel correction coils, of the flipper correction coils and of the flipper themselves.

Neutron tests on instrument SN3 have shown that the high surface quality mirrors (a few  $\text{Å}$  peak to peak) necessary for optical focussing are available. They have been developed for advanced X-ray telescopes. However, magnetic coating of such a surface seems to give a small angle scattering contribution. Further test will be made.

The definition of the choppers for the time of flight option has been completed (ILL tech.rep. 87KO10T). The electronics is being defined at KFA-Jülich.

## D7: Diffuse scattering instrument with polarization analysis

Modifications carried out in 1987 were directed towards reliability tests of the analyzer transmission and flipping ratio as a function of wavelength, and to facilitate an easy transition between the instrument version with spin analysis and the version without analysis. Also, the resolution limits were improved.

The instrument is now equipped with 28 double coated low curvature analyzers with 200 mirrors each, i.e. 8 analyzers more since last year. The production of one such analyzer needs an average time of 1 month work of evaporation. Only 4 further analyzers are still equipped with 50 one-side coated mirrors of 1 mm distance and show thus the poorer behaviour in the  $\lambda$ -dependence of transmission and flipping ratio.

All four banks are now equipped with pneumatic jacks. Measurements of transmission as a function of  $\lambda$  in March and August showed that with this improvement on all four banks the  $\lambda$ -dependence of the transmission is fully reproducible for all analyzers, on the condition that the incident wavelength is not changed during that time. (We restricted ourselves to  $\lambda = 4.8 \text{ \AA}$ ). We have started to take measures to overcome the fixed-wavelength limitation. The main problem to be solved is that different polarizer curvatures are needed for different wavelengths. The sample axis has to be shifted according to the curvature so that the chopper axis, the sample axis and the collimation slit always remain aligned in the maximum neutron intensity direction.

The final aim is to avoid remeasuring the  $\lambda$ -dependence of the analyzer transmission for each experiment by using a transmission table in the computer determined once. For the measurements from March to August it is already possible to do this. But afterwards the wavelength was changed to  $3.1 \text{ \AA}$  for an experiment without polarization analysis. One of the next steps is to compare the transmission again if one goes back to  $4.8 \text{ \AA}$ .

The curvature of the new analyzers changes during the course of time. The curvature screws have to be tightened from time to time to avoid the transmission of neutrons without being reflected, i.e. "direct sight".

The flipping ratio i.e. the polarizer efficiency is constant with  $\lambda$  over the total range of transmission, whereas with the older analyzer version it was a function of the wavelength because the shorter wavelengths were not absorbed well enough.

The collimator boxes can now be shifted below the analyzers and it is thus possible to switch very quickly from the spin analyzer version to the version without spin analysis (in 1/2 hour).

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

D11 has operated routinely without major incident throughout the year. In February a major effort was made to eliminate electrical parasites which had been perturbing the data acquisition system. This was done by connecting all mechanical and electronic components to the same earth and filtering the main sources of noise. The work was highly successful and data acquisition is now totally reliable. Other improvements for increasing the operational reliability of the instrument were the

purchase of a third pumping group and a spare cooling system for the detector electronics.

A new power supply has been purchased for the velocity selector "Brunhilde". This has been installed and tested under manual operation and will soon be adapted to computer control.

A special bow has been built for the horizontal sample changer enabling it to work in a controlled dry atmosphere facilitating experiments carried out at  $\sim 0^\circ\text{C}$ .

The principal change in data treatment facilities has been the purchase and installation of a colour display system which allows the user to display as a two-dimensional array either previously measured data or that currently being measured. This is particularly useful in the case of anisotropic spectra. Some examples may be found in the report of College 9b.

## D17: Low-Q, high resolution diffractometer on cold guide H16

This year has seen a full experimental program on D17 which has once again demonstrated the versatility of this small angle, long wavelength spectrometer. Although a large part of the work consists of small angle scattering experiments, a wide variety of other measurements are made. These have included reflection studies, time-of-flight inelastic scattering and diffraction measurements. In some experiments polarized neutrons have been used. The plethora of different configurations and need to change rapidly from one mode to another has sometimes placed a strain on the technical services of the Institut which have, in general, responded rapidly and efficiently to many varied demands.

An accident at the end of January in which the helical slot rotating velocity selector left its bearings and hit the casemate wall brought forward a general review of replacement and new selectors. Despite the occurrence at a week-end, the defective selector was removed and replaced with an alternate selector within 12 hours. The technicians worked extremely hard to provide a reasonable alternative of high and low resolution selectors within a few days while repairs were made to the badly damaged selector.

Unfortunately it has not proved possible to refurbish the selector to the full specified performance and a new selector is now at an advanced stage of construction. This will allow the ILL to test new materials for the construction of the drum and a design with plastic composite lamellae has been agreed. It is hoped to test the new selector very early in the New Year before decisions are made on other new selectors required by the Institute.

The general technical development has not been neglected. A simple colour display installed in April has proved popular with users particularly those studying anisotropic samples. Some examples of the contour plots are shown elsewhere in the report (College 9). Several improvements made to the control program to simplify step motor control for reflection measurements and automatic control of the cryostat temperature. As previously all these modifications are also immediately applicable to D11. At the time of writing, a new cryostat for small angle scattering is undergoing cryogenic tests. The IEEE 488 interface is also under test prior to installations on the instrument with multi-meters and other equipment for monitoring and control of sample environments.

## D22: A new low-Q diffractometer on the Horizontal Cold Source

Following detailed discussions, a simplified version of the small-angle scattering instrument D22 has been proposed to the Instrument Subcommittee in September 1987. On this occasion, the project was endorsed. D22 will be a "classical", i.e. a long D11-type, instrument easy to use. An increase of the Q-range covered in any single measurement is achieved by a lateral displacement of the detector. The time-of-flight (TOF) and the polarized neutrons options are postponed for later development, simultaneous detection of large and very small Q can be realized by a special sample zone set-up.

Prior to the final decision of the Instrument Subcommittee, the definition of the detailed concept of D22 had started with the preparation of the call for tenders for the detector tube offering minimal and maximal sample-to-detector distances of 1.2 m to 18 m, respectively. Of the three options studied, the one with a tube diameter of 2.5 m, able to house a laterally displaceable 1 m by 1 m detector being developed by the ILL detector group, has been adopted by the Subcommittee. The detector tube is to be built of stainless steel, not for the benefit of D22 itself, but in order to provide a clean magnetic environment for its neighbours, especially the m experiment. The order for the detector tube and mechanics has been placed before the end of 1987; the installation is planned for early 1989.

The specifications and a tender document for the second largest bit of the instrument, i.e. the collimation unit, are finished as well. Offers are expected by the end of February, 1988. The definition of the other mechanical parts will be based on the design for D11 and D17 (sample environment) or has to wait for the completion of other preparative work, like the construction of the selector.

Coordinators: A. Dianoux  
R. May

## 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 (C. Ritter, J.L. Soubeyroux, K. Ben Saidane).
- D2B : Very high resolution powder diffractometer on thermal beam H11 (A. Hewat, J. Davies).
- D3B : Two-axis polarized neutron diffractometer with lifting counter on thermal beam H5 (F. Tasset, J. Baruchel, A. Dorn).
- D4B : Disordered materials diffractometer sharing the hot beam H8 with IN1B (P. Chieux, A. Barnes, A. Hawes).
- D9 : Four-circle diffractometer on the 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, G. Schmid).
- D16 : Four-circle MK6 diffractometer on cold guide H16 (G. Zaccari, L. Braganza, E. Pebay-Peroula, J.R. Reynal).
- D19B : Multidetector diffractometer for protein crystallography (S.A. Mason, J. Chevrier, M. Berneron, J. Archer, D. Robinson).
- D20 : High flux multidetector (J. Pannetier, P. Convert, J. Torregrossa).
- DB21 : Four-circle diffractometer with PSD for biological macromolecules (M. Roth, P. Metcalf (EMBL), P. Agnes).
- S18 : Neutron interferometer on the thermal neutron guide H25 (A. Rumpf, G. Schmid).
- S20 : Neutron topography diffractometer (J. Baruchel).
- S21 : Double crystal diffractometer (C. Zeyen, R. Chagnon).
- S42A : Laue Diffractometer (J. Marmeggi, W. Drexel).
- S42B : Laue Diffractometer (J. Baruchel, A. Filhol).

## Major Instrument Modifications

### D1A: High Resolution Neutron Powder Diffractometer on the H22 Guide

D1A played an important part in ILL experiments on high  $T_c$  superconductors in 1987, because, as a special instrument, it is less tightly scheduled than D2B. The solution of the low temperature structure of superconducting  $\text{YBaCu}_3\text{O}_7$  by Capponi et al. (see elsewhere) is one of the most cited papers in the field.

Other important superconductor experiments included the magnetic structure of  $\text{GdBaCu}_3\text{O}_7$  by Paul et al., and sam-

ple characterisation for many other groups. Apparently D1A still has advantages over pulsed source machines because of the ease with which low temperatures can be programmed, and because of the long wavelengths available at low scattering angles, essential for magnetic work, and for the study of large structures, such as zeolites.

Because of the heavy overload on D2B, D1A was also used for some scheduled experiments, including work on stress in engineering materials. Some D1A beam time was also sold to an industrial group for this purpose, and other time was exchanged in return for electronic components, programming and technical help.

As a special instrument D1A has no official ILL financial or manpower support. Of note is the assistance provided by Professors H. Fuess, and A. Simon in seconding staff to ILL (J. Cockcroft and T. Vogt) in return for D1A beam time.

## **D1B: Two-axis Diffractometer on Thermal Guide H22**

87 has been an exceptionally busy year for the D1B powder diffractometer. The shutdown during February/March was used to replace the old Solar computer (The last of his kind working at the ILL) by a more standard PDP 11/34. This complete system change required of course the replacement of the major parts of the experimental set-up (electronic hardware and software), including the motor driving and controlling modules and coders, the data acquisition cards and the instrument inherent data display system. Due to the good efficiency of the electronic and the informatics department this quasi rebuilding of the diffractometer needed only 2 weeks of off-beam-time and just one week of the precious beam test time to be successfully completed.

The program MAD used on most diffraction group instruments is working now on D1B hereby facilitating the use by visitors. The implemented PARAME program allows the easy handling of the MAD data acquisition system. Data being transferred as well to the DEC 10W as to the central VAX computer, the data evaluation programs were modified to allow for the new format.

A second change to the more powerful PDP11/24 was carried out in September. The available data storage memory on disc has increased, the automatic internal disc change allows to follow fast kinetic experiments with very high data acquisition rates without bothering about a necessary disc exchange. A variety of interesting experiments ranging from the study of the kinetics of cement hydration under autoclave conditions, the kinetics of the dehydration of clay minerals in situ, investigation of structural transformations of an electrode during charge/discharge, phase transitions in general, up to the "normal" structural experiments on e.g. order of intercalated guest atoms in a host lattice at different temperatures and stoichiometries or magnetic wave vectors at very low temperatures were successfully completed (See College 5 : "Highlights" for the option of polarised neutrons).

## **D2B: Very High Resolution Neutron Powder Diffractometer on Beam H11**

D2B has been heavily scheduled in 1987 to make up for time lost due to initial mechanical problems. These problems have been resolved with the support of the large detector bank on air pads. Detector positioning is very precise, due to the direct

drive axial encoder, but is still too slow. This remaining problem will soon be resolved.

The machine has been most heavily used in its high flux mode, which allows resolution similar to D1A, but with data collection typically 60 minutes or less per scan. In particular it has been used for temperature dependent studies of superconductors, refinement and comparison of many different zeolite samples etc. Because of the good resolution at low scattering angles, it has been particularly successful for magnetic structures, such as that of  $\text{Pu}_2\text{O}_3$  by Lander et al. where the earlier pulsed neutron result was corrected.

The ability to work at short wavelengths (1.05Å or less) and scattering angles to 165° is also an advantage for very precise structural studies, such as the resolution of oxygen disorder in the superconductor  $\text{YBaCu}_3\text{O}_7$ . This work was the first in collaboration with Swiss scientists since Switzerland officially joined ILL.

There are still two areas for improvement: the monochromator, which gives a shoulder on low angle peaks in the high resolution mode, is being replaced, and a graphite filter is needed to permit routine operation at wavelengths of 2.4Å and higher.

## **D3B: New Polarized Neutron Diffractometer**

Early in 1977, D3 had to be removed from the thermal beam tube H5 for being replaced by the new horizontal cold source. Since then D3B, an extensively overhauled and modernized version of D3 has been successfully installed on the hot neutron beam tube H4 finally occupied by D5.

The primary axis features a state of the art polarising spectrometer system for short wavelength neutrons comprising the following important elements:

- A magnetic monochromator cassette system for the addition and exchange of new and activated polarising crystals.
- An in-shield Carousel system for insertion of higher order resonance filters, secondary beam shutting with a dual rotor assembly including guide fields, and beam monitoring with a vanadium foil.
- A non dispersive cryoflipper to control the incident polarisation.

The original biological shield of D5 has been reused except for the system to drive the take-off angle which has been completely rebuilt. To improve the convenience and flexibility the secondary spectrometer is now placed on a Tanzboden floor. The D3-Harwell MKVI normal-beam diffractometer has been modified slightly to cope with this new position. It is now supported by a heavy-duty, low height, air cushion base plate.

The beam aperture and sample positioning is fully controlled by a new motorised non-magnetic slit system interfaced to the computer. A commercial, non magnetic xyz manual translation system is supporting the cryostat. The successful D3 pneumatic half-shutter system remains in front of the single  $\text{He}^3$  detector for precise orientation matrix determination. Additionally, the cryomagnet field (0 to 4.6 Telsa) and sample temperature (1.5 - 300 K) will be fully computer controlled.

A new PDP 11/73 compact computer configuration including magnetic tape streamer and hard disk memory is now being installed. It should permit efficient and trouble free data acquisition on this sensitive, high precision instrument with high counting rates.

The installation had been enhanced with respect to the initial schedule in order to test some essential neutron characteristics before the reactor shut down in November 87. They have been found satisfactory and D3B will be routinely scheduled one month after reactor start up early in 1988.

### D4: Disordered Materials Diffractometer on the Hot Source

The instrument had been working on the standard configuration i.e. with two multidetectors placed respectively at 1.50 m and 0.75 m from the sample, for most of the time. This has allowed us to perform without difficulty several experiments at the limit of the present possibilities of the machine, i.e. with a statistical accuracy of 1%. The demand for this high accuracy and stability is slowly increasing and a systematic control of the stability with a repetitive scan technique is now routinely achieved. It implies an increase of the data collection rate and we have therefore tried to install a Winchester drive unit to reduce the turnover of our data disks. However the practice which has finally emerged is to perform the data regroupment and display on the VAX with an accurate laser print output for the graphs. It allows us to quickly and precisely assess the quality of the experiment, to identify background problem, etc. The data handling programs installed on the VAX are moreover easy to be developed and compatible with the "Liquid" programs written for D20. Of course, in the long term, the philosophy of the data handling and evaluation on the D4 machine itself needs to be reconsidered.

A large variety of sample environments have been used during the year (high temperature, low temperature) but we should especially mention several measurements under pressure with the test of various pressure vessels up to 1.5 kilobars. The accuracy of the measurements has allowed us to further investigate the problems of background and beam intensity profile which should in the near future improve our understanding of container and sample environment corrections.

The 3-meter detector distance has been installed on several occasions and has been proved quite useful for low angle measurements (down to about  $1^\circ(2\theta)$ ) and improved resolution (e.g. for low temperature magnetic structure investigations).

### D9: Four-Circle Diffractometer on Hot Beam H3

D9 has operated very smoothly, and because of the increased flux larger, organic systems are now also studied, often at low temperatures. A prototype of a new horizontally focusing Be-monochromator was tested, but final installation depends on the preparation of a full set of Be microcrystals. The major improvement was the change-over from the 9-year old PDP11/34 to a Microvax II. This will facilitate life for the experimentalist, as he can now use the same operational language on the instrument and the central computer. Another improvement dictated by the higher flux, were modifications in the measurement procedures which now allow the recording of up to 1000 reflections a day with neutron detection efficiencies of 90 % or more.

### D10: Four-circle Triple-Axis Spectrometer on Thermal Guide H24

This year D10 has again been performing a variety of mostly three-axis experiments for colleges 4, 5, and 6.

Often used for high energy and momentum resolution, it may be useful to recall two favourable configurations.

For  $k_1 = 2.66 \text{ \AA}^{-1}$ , Cu(200).10'.10', PG(004), 40' gives a vanadium energy width of .04THz and a momentum resolution of  $Q_{//} \sim Q_{\perp} \cong .003 \text{ \AA}^{-1}$ .

For  $k_1 = 1.55 \text{ \AA}^{-1}$ , PG(002), 10', 10', PG(002), 40' yields an energy resolution of 0.2THz and similar Q resolutions.

During 1988 we expect our new 4-circle cryofurnace (Pujol, Zeyen) to be operational. This device is designed to reach less than 2 K sample temperature, at lower He consumption than our previous cryostat successfully operated for nearly ten years now. Due to the particular design of the inner seals and clamps, sample temperatures up to 600 K will be possible. BENOIT (CNRS GRENOBLE) is furthermore developing a dilution option which tested on a 4-circle simulation device has already reached .125 K.

### D15: Four-circle MK6 Diffractometer on the Inclined Thermal Beam IH4

D15 has been scheduled regularly throughout the year for experiments mostly concerned with the effect of external constraints on nuclear and magnetic structures. The constraints applied have been magnetic fields of up to 4.6 T, pressures up to 30 kbars and temperatures in the range 50 mK - 1800 K. The figure 106 compares the magnetic (H,T) phase diagrams of  $\text{Eu}(\text{As}_{0.9}\text{P}_{0.10})_3$  and  $\text{Eu}(\text{As}_{0.20}\text{P}_{0.80})_3$  which have been determined.

Development of the small multidetector for D15 of  $32 \times 32$  pixels ( $8 \times 8^\circ$ ) continues, but an acceptable degree of uniformity and stability has not yet been obtained.

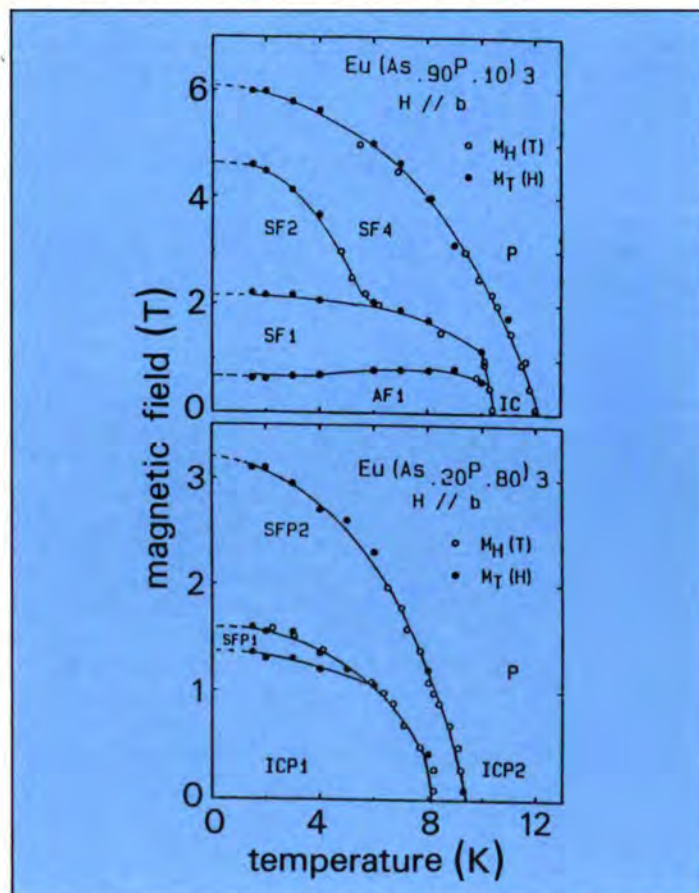


Figure 106: Magnetic (H,T) phase diagrams of  $\text{Eu}(\text{As}_{0.90}\text{P}_{0.10})_3$  and  $\text{Eu}(\text{As}_{0.20}\text{P}_{0.80})_3$

## D16: Four-circle MK6 Diffractometer on Cold Guide H16

The new monochromator housing is being installed during the reactor shutdown at the end of 1987. By having the beryllium filter inside the housing, so that the first defining slit is considerably closer to the monochromator than in the present set-up, collimation geometry conditions should be improved significantly to result in higher incident beam intensity at a given resolution. The new configuration will also have the possibility of bringing the sample axis closer to the monochromator, thereby allowing the full available beam divergence to fall on the sample in experiments that can tolerate the relaxed collimation conditions.

The next instrument improvements planned concern the replacement of the PDP-11 computers, which are now old and not as reliable as could be wished, with a micro-Vax system. This will also make D16 fully compatible, for data collection and analysis, with the other diffractometers and small angle cameras with which it is scientifically complementary.

## D19: Multidetector Diffractometer for Protein Crystallography on H11

The  $4^\circ \times 64^\circ$  position sensitive detector (p.s.d.) on D19 functioned perfectly throughout 1987 as did the 2-stage Displex cryorefrigerator. For certain low-temperature experiments where scattering from the outer A1 heat shield interferes with reflections from the sample, a new Vanadium heat shield will be available.

Background due to stray neutrons from neighbouring instruments is no longer present as the D19 zone has been isolated using flexible B4C curtains. All background entering the detector is now caused by D19 itself.

One of the two alternative D19 Soller collimators which had been used for some 2 months in the unmonochromated H11 primary beam (i.e. before the monochromator) was found to have collapsed: the blades were of Gd-coated mylar. Although it is not certain that this collapse was due to radiation damage, a new collimator at the IN8 design with Gd-painted Al blades is now under test.

Motorisation of the graphite filter is under way. And the possibility of moving the detector closer to the sample is under investigation, to increase the region of reciprocal space seen per frame. However this is at best a stop-gap solution which would introduce geometric distortions and parallax for very limited gains.

Experiments on small and medium-sized molecules, using the intensity-integration procedures of Wilkinson, Stansfield and McIntyre, make use of the p.s.d. in different ways. For unit cell volumes below about  $2000 \text{ \AA}^3$  it is often convenient to measure reflections in the equatorial plane only, while for larger unit cells, normal-beam scans making fuller use of the p.s.d. are appropriate.

The limited processing power of the VAX11/750 has slowed down significantly a number of experiments. Plans are in hand to replace the PDP11-VAX combination by a single more powerful processor. All raw data are now archived on tape after typically 5-fold compression. The long-term aim is archived on high-density storage devices on the ILL central computer as for other ILL instruments.

Several versatile display and plotting programs for D19 data are now available on the DIVA central computer.

Experiments on DNA fibres under controlled humidity conditions provided a demanding test on the p.s.d., as a full 2-dimensional diffraction pattern can only be built up by juxtaposing  $4^\circ \times 64^\circ$  frames (each requiring 2 hours) measured at, say,  $2^\circ$  intervals in detector rotation.

The experiments on human haemoglobin and on DNA fibres, although promising, have shown the urgent need for a 10-fold increase in detecting solid angle. This would be possible if 3 or more vertically-curved detectors, each  $20^\circ$  wide and  $64^\circ$  high, were constructed. An alternative design with 4 or more  $20^\circ \times 20^\circ$  flat p.s.d.'s would be a less satisfactory solution. Either design would facilitate also experiments on liquid crystals, intercalates, etc. where the spectra of interest have features much broader than the maximum of  $4^\circ$  available on the present detector.

## D20: High Flux Multidetector Diffractometer on Beam H11

D20 has started normal operation during this year and has been regularly scheduled during the last two reactor cycles of 1987. Owing to the small angular coverage of the current detector ( $12.6^\circ$ ) experiments were mostly concerned with liquid or amorphous materials and performed in the scanning detector mode with the shortest wavelengths available ( $\lambda = 0.9$  or  $1.3 \text{ \AA}$ ). The use of the longest wavelength ( $\lambda = 2.4 \text{ \AA}$ ) is still hampered by the limited supply of graphite filters which forces to use only a part of the beam.

Mechanical problems in  $2\Theta$  and  $\omega$  positioning have been experienced during the recent operation and are expected to be resolved soon.

The software for data evaluation, largely based on D1B and D4 software, has been developed for use on DIVA.

Co-ordinator : J. Pannetier



General view of the D20 diffractometer.

# SPECIAL INSTRUMENTS AND EXPERIMENTS

(W. Drexel)

Special beam activities have developed out of the basic idea of the ILL to supply neutrons to all scientists that need them for their research work (H. Maier-Leibnitz, B. Jacrot, BIST 165). ILL has therefore grouped together in this area those activities that are complementary to the routinely scheduled normal instruments:

a) experiments that need dedicated equipment such as the search for an electrical dipole moment of the neutron, the search for neutron-antineutron oscillations, neutron lifetime measurements or the precision measurement of  $h/m$  etc.),

b) development of new scientific equipment (such as supermirrors) or c) development and improvement of novel experimental and instrumental methods and their first applications (such as strong VCN-UCN sources, neV-resolution diffractometry and spectrometry, simultaneous use of neutrons and  $\gamma$ -beams in diffractometry, neutron topography or new projects such as VCN-interferometry and grazing incident/exit diffractometry for surface studies). - Many of these activities are financed and supported by external groups, some are ILL activities or collaborations. - All proposals for S-activities are treated by the appropriate Subcommittee and are revised periodically. The important ones are covered by contracts.

The table shows an overview of actual SB-activities at the ILL. Compared with the list of 1986 one can see that the following activities have come to a close:

- 1) S44 (channeling, blocking); HMI, Berlin
- 2) S52, neutron lifetime measurement, counting the decay of neutrons in a beam (LAPP, ISN, ILL)
- 3) the neutron charge measurement (Bayreuth, Munich)
- 4) the mass separator OSTIS (SN6) is running out and
- 5) the development of a neutron microscope is interrupted during the development of an appropriate position sensitive detector.

## SB-EXPERIMENTS/INSTRUMENTS:

S10	(n, $\alpha$ reactions)	Gent/Mol
S18	neutron interferometer	Dortmund/Wien
S20	neutron topography	CNRS Grenoble
S21	high resolution double crystal spectrometer using neutrons and a $\gamma$ -beam simultaneously	ILL
S30	concentration profiles by (n,p),(n, $\alpha$ )	HMI Berlin
S34	$\gamma$ - $\gamma$ correlations	ILL/Sussex
S42	Laue diffraction	CNRS Grenoble
S50	$h/m$ determination	Braunschweig
S51	fission, neutron activation analysis	Darmstadt/Grenoble
GAMS4	double flat $\gamma$ -crystal spectrometer	NBS/ILL
SN5	UCN-source on level C	ILL
SN7	beam of polarized neutrons	ILL
H17	cold beam	ILL
H18	cold beam	ILL
S-UCN	superthermal liquid He-source	Rutherford/Berlin
nn	neutron-antineutron oscillations	Heidelberg/Padova
TGV+ } Turbine)	VCN-UCN-source on level D	ILL
NESTOR	magnetic bottle for neutron lifetime	Bonn
EDM	neutron electric dipole moment	Sussex et al.
n-bottle	neutron lifetime experiment in a neutron bottle with variable volume	ILL
UCN	gravity diffractometer	TU Munich/ILL
externally supported Instruments: IN3, DIA		
Under construction: NESSIE neutron gravity spectrometer		
Projects:	VCN-interferometer	Vienna/TU Munich
	Grazing Incident/Exit Diffractometer	Univ. Munich

Important progress in the area of SB-activities during 1987 can be summarized as follows (see also the Instrumental Section of College 3 for reports concerning nuclear and fundamental physics and College Secretaries' reports for scientific results):

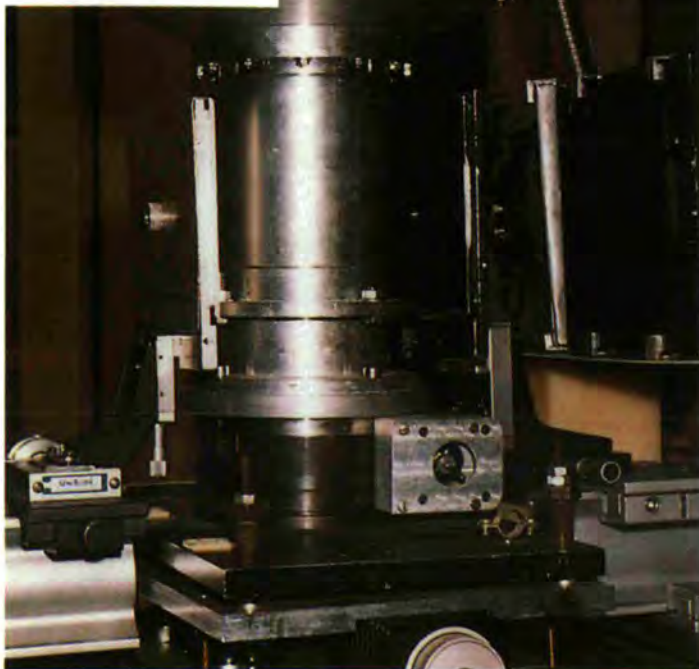
**S18**, the neutron interferometer, tested new interferometers ( $\pi/2$  interferometer, 4-plate-interferometer, two crystal interferometer, interferometry at variable wavelengths). First time-resolved interferometric measurements have been performed using a chopper in front of the interferometer. The mechanical set-up has been reinforced so that heavier equipment can be used now.

**S20**, neutron diffraction topography, is under reconstruction. The neutron flux will be increased and S20 will be better adapted to proposed experiments (magnetic phase transitions, test of extinction theories, memory effects of domain structures, "local" investigation of crystal properties...). An important improvement was achieved in 1987 by a semi-automatic control of scans and polarization, and by the use of a high resolution PSD ( $\approx 0.2\text{mm}$ ) developed at the LAPP, Annecy. The new instrument will allow to change the incident wavelength more easily, to record Bragg spots which are out of the horizontal plane; overall reliability is expected to improve too. It is planned to adapt an ILL standard T-controller and to install an automatic control of the applied magnetic field. The reconstruction is expected to be finished early next year.

**S21**, the high resolution double crystal spectrometer with an on-line  $\gamma$ -ray diffractometer, has been operated successfully for a number of user experiments this year taking full advantage of its new intense beam on H25. Coupled neutron and  $\gamma$ -ray diffraction, first performed on S21 this year, revealed the power of this technique. Around the tricritical point of KDP, applied electric field measurements yielded results in conformity with Landau theory, wiping out speculations based on standard diffraction techniques about the existence of higher order terms. Applied to the incommensurate phase in

Quartz, this new technique allowed to demonstrate for the first time the existence of single and triple-q states in unstressed crystals. High resolution small-angle scattering experiments at resolutions far beyond D11 ( $10^{-3}$  to  $10^{-5}$  Å<sup>-1</sup>) were shown to be possible with reasonably good intensity. Further progress with background reduction using perfect Si (331) channel-cut crystals has to be made next year to make the method competitive.

**S42**, the Laue diffractometer, got new back neutron cameras receiving films  $13 \times 18$  and  $24 \times 30$  cm<sup>2</sup> with Li-ZnS(Ag) converters. The instrument has been slightly modified to accept the dilution refrigerator that can be mounted on an orienting device ( $\pm 3^\circ$  tilt,  $\pm 180^\circ$  rotation). The crystals are attached to a copper sample holder and then mounted on a goniometer head for pre-orientation. Symmetric and unsymmetric Laue patterns could be recorded above and below magnetic transition temperatures as low as 0.5 K. S42 can accept a special furnace that allows in situ crystal growth (up to 1750 K) and high T neutron diffraction experiments. It is thus possible to study polychromatic neutron diffraction from  $0.3\text{K} < T < 1750\text{K}$  with S42. (See photographs).



S42: top: insert and dilution unit used to cool the samples down to 0.3 K attached to the goniometer head fixed to the bottom of the loading probe. bottom: view of the dilution refrigerator mounted on the orienting device of the S42 Laue diffractometer.

**S50**, the  $h/m$  measurement, has received its final apparatus. It was installed and aligned at the end of 1987. (See photograph).



S50: It is the aim of S50 to determine  $h/m$  ( $h$ : Planck constant,  $m$ : neutron mass) to an uncertainty of  $10^{-6}$ . As  $h/m = \lambda/v$ , one has to measure the wavelength and the velocity of the same neutrons. In S50, a beam of neutrons of well-known wavelength is produced by back-reflection on a perfect Si-crystal. The velocity is measured by a refined time-of-flight method (using polarized neutrons, phase modulation and interference patterns). The photograph shows the vacuum vessel of about 20 m length containing the experimental equipment for the determination of  $h/m$ .

**GAMS4**, the double flat crystal spectrometer, was substantially improved in 1987. Its gamma-ray detector can now be rotated and translated. The NaI detector was replaced with an intrinsic Ge detector. GAMS4 got a data acquisition system similar to the one in use on GAMS2/3. Great effort was put into the temperature control system as T variations are one of the most serious problems in sub-ppm measurements. A sealed environmental enclosure around the spectrometer has been installed and can be filled with any gaz. Four temperatures are recorded regularly (crystal, interferometer arm, instrument, air) and the temperature of the instrument can be controlled within 1/100 of a degree. Major improvements have been achieved in the instrument software: the instrument can recover from numerous fault conditions, a utility program for easy aligning, testing and tuning was made available as well as programs for rapid but thorough analysis of GAMS4 spectra. The instrument is considered to be ready for lifetime and structure factor measurements, for high precision wavelength measurements some more improvements are necessary.

**SN5**, the VCN-UCN source on level C, saw its experimental area reduced in 1987 due to the installation of the horizontal cold source. The remaining two beam positions have been used for preparatory experiments and development work (e.g. UCN-monochromator).

**SN7**, the polarized neutron beam, was fully used as in 1987. It was equipped with a thermo-isolated cabine to improve instrumental stability and working conditions.

**H17**, Neutron flux and TOF spectra measurements have been performed before the liquid He UCN source was reinstalled, shielding against background has been improved so that the TOF experiment on upscattering of neutrons in superfluid He was able to be performed.

**H18**, the 10m optical bench from the neutron charge experiment was left in place for several neutron optical test measurements.

nñ, construction work for this huge experiment continued in 1987. The inauguration of the detector house was celebrated.

**TGV + turbine**, the new UCN-VCN source on level D was in routine operation in 1987 and supplied neutrons to the following experiments: EDM, NESTOR, n-bottle and a neutron microscope that was replaced in June with a gravity diffractometer, an experiment to look for deviations from the  $1/v$  law for the absorption cross-section in U at very low values of  $v$  and some more test experiments. The  $\gamma$ -background from neutrons captured in the neutron guide, the turbine and its housing was reduced by installing lead shields at some critical points; it is, however, still a little too high for cases when permanent presence is necessary (e.g. for the installation of new experimental equipment). The neutron flux from the vertical guide has shown to be fairly stable over the last year. -In June 1987 we started the installation of the neutron gravity spectrometer NESSIE from the reactor of Garching. First the turbine exit was equipped with its neutron guide of enormously complicated geometry in order to feed the NESSIE monochromator in an optimal way (it is curved in three dimensions !). Between June and October the neutron lifetime measurement with variable storage volume profited from the high intensity at this position. In Oct.-Nov. 1987 the monochromator of NESSIE was installed; the analyzer is scheduled for March 1988.

**Projects:** Ideas for improvements exist for nearly all SB-experiments and many of them can certainly be realized. There are 2 main new projects that will be started at the ILL in 1988:

1) The VCN-interferometer (Vienna/TU Munich) on the VCN exit on level D. Two important steps were made in 1987 by the Vienna group in solving the vibration damping and the financial problems.

2) A project of the University of Munich aiming at the construction of a grazing incident/exit diffractometer for surface studies.

## SPECIAL STUDIES OF EXPERIMENTAL AND INSTRUMENTAL TECHNIQUES

### Experimental Work (P. Ageron, A. Beynet)

This concerns mainly the performances of some components and of the whole of the horizontal Cold Source:

a) A major task was the qualification of the surfaces and of the  $^{58}\text{Ni}$  coating for the final mirrors (glass or metal) before assembly.

The goniometer on PN5, with a monochromatic beam of about  $60 \text{ \AA}$  permits the measurement of the reflectivity and the critical angle of a pair of mirrors (500 mm long, 40 mm of useful height, 6 mm distance) by rotating from minus to plus the critical angle.

b) Another major activity were the preliminary measurement of the neutron brightness of the new Horizontal Cold Source from its first start to full reactor power. A collimation basis of two slots (3 mm wide, 1 to 10 mm high, 2m apart) placed behind the safety valve, allows for the observation of either the top or the middle of the cold cell. Either a block of 10 cm Be at room temperature or a chopper with a slot of 3 mm wide on a circumference of about 1 m at a constant speed of 1450 rpm can be placed into the beam. In both cases the detector is a fission chamber of low efficiency ( $\sim 2.10^{-3}$  at  $1 \text{ \AA}$ ).

c) The observation of the top of the cell (the full beam being filtered by the Be) permits the detection of a lack of liquid  $\text{D}_2$  in the zone: at 30 MW, when an artificial lowering of the  $\text{D}_2$  level was induced, the neutron counting rate showed a reduction from 390 to 130 c/s. Up to the full power of 57 MW no lowering of the liquid  $\text{D}_2$  level was observed under normal operational conditions. From 6 to 57 MW, however, a slight decrease (17 %) of the neutron counting rate (normalised at full power) has been observed. This could indicate that larger bubbles emerge at high reactor power.

d) "Brightness" spectra (without reflexion on the guides) have been measured at the centre of the source with the chopper by using a multi-channel analyser of 512 channels of  $80 \mu$  width. The variation of the channel width from 70 to  $100 \mu$ s does not influence the results. A high background ( $\sim 25\%$  of the peak) reveals only significant results between about 1 and  $10 \text{ \AA}$ . The results are summarized (normalized at full power) on Fig. 107.

The spectrum A is measured at 56 MW, spectrum B at 6 MW. As one expects, the latter is colder than spectrum A. The calculated spectrum C (with 20 % vacuum, but for a smaller (18 cm) diameter instead of that of the final cell (21 cm)) is higher than the measured one in the thermal region ( $1-2 \text{ \AA}$ ) and lower in the cold region ( $2-10 \text{ \AA}$ ).

These first results give qualitative information. New measurements with guides and better normalization are required in order to assess the full performance of the new Cold Source.

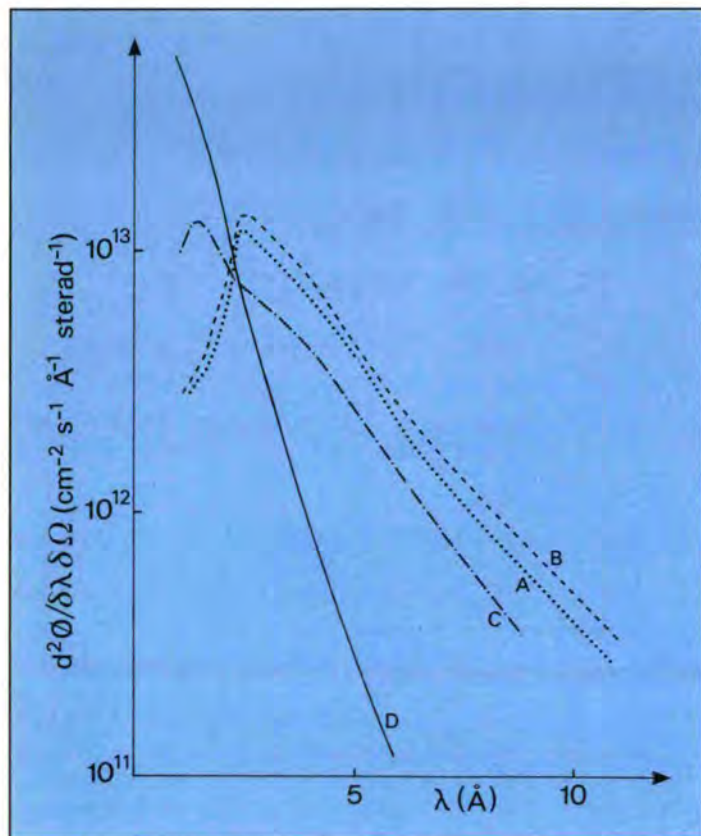


Figure 107: Brightness spectrum at the centre of the horizontal cold source of the HFR (provisional results)  
**Measurements:** A at 57MW, B at 6 MW  
**Calculations:** C calculation by DOT for a smaller cell ( $\varnothing$  18cm instead of 21cm);  
 20% vapour contents  
 D theoretical maxwellian spectrum at 320K and total flux  $8 \cdot 10^{14} \text{cm}^{-2} \text{s}^{-1}$  (all normalized at 57 MW)

## Calculations (A. Robert)

### a) Transmission of the guide H511

The programs TG1.F4 and TG3FD.F4 have permitted to recalculate the transmission of the guide H511 (with Fe-Co coating), by taking into account that of the preceding guides and of the bender; thus allowing for a better specification of the source of neutrons to be focussed (see next paragraph).

### b) Focussing of slow neutrons ( $\lambda > 10 \text{ \AA}$ ) for IN15

P is considered as a neutron emitting point at the exit of the H511 guide and we have tried to obtain an image of P at a point P' being 20 m further away, by using a focussing device being placed at equal distances between P and P'. The following facilities were envisaged:

#### - Cryogenic hexapole (magnetic focussing)

The response of this device has been studied analytically as well as by simulation (program HXPT.F4) (see report 87R006T). The yield is limited by the necessity of diaphragm the beam in order to achieve acceptable focussing. By supposing a forward emission of 1 neutron/sec.cm<sup>2</sup>.ster. in the plane of P, one receives about  $4.25 \cdot 10^{-5}$  neutrons/sec. in a small surface around P'.

#### - Guides and focussing mirrors

The shape of their reflecting surfaces are always produced by ellipsoids with focal points P and P'. For that reason one could consider a guide of squared or circular section or even a super-mirror with the shape of a "chistera" inducing a deflection. Their yield (neutrons in P') is increasing in that order and can reach 10 times that of the hexapole, but their geometrical form is a major obstacle to their realisation. The final choice has not been made.

### c) Hydrogen doping of the Horizontal Cold Source

We have calculated (program DOT3) the gain variation of cold neutrons as a function of a small H concentration in the deuterium of the Cold Source. For the present geometry of the Cold Source the H content (1/2 ortho, 1/2 para) in the deuterium has been varied from 0 to 40 %. The optimum gain (about 20 %) has been obtained for a H content of about 10 %.



**DIRECTORATE SERVICES**

# SCIENTIFIC COORDINATION AND PUBLIC RELATIONS

## SCIENTIFIC PROGRAMME IN 1987

The reactor operated routinely during the five cycles scheduled in 1987 (refer to Reactor Department section in this report). Due to the installation of the new Cold Source the number of cycles was reduced by one cycle - there was a shut-down from middle of November to the beginning of January.

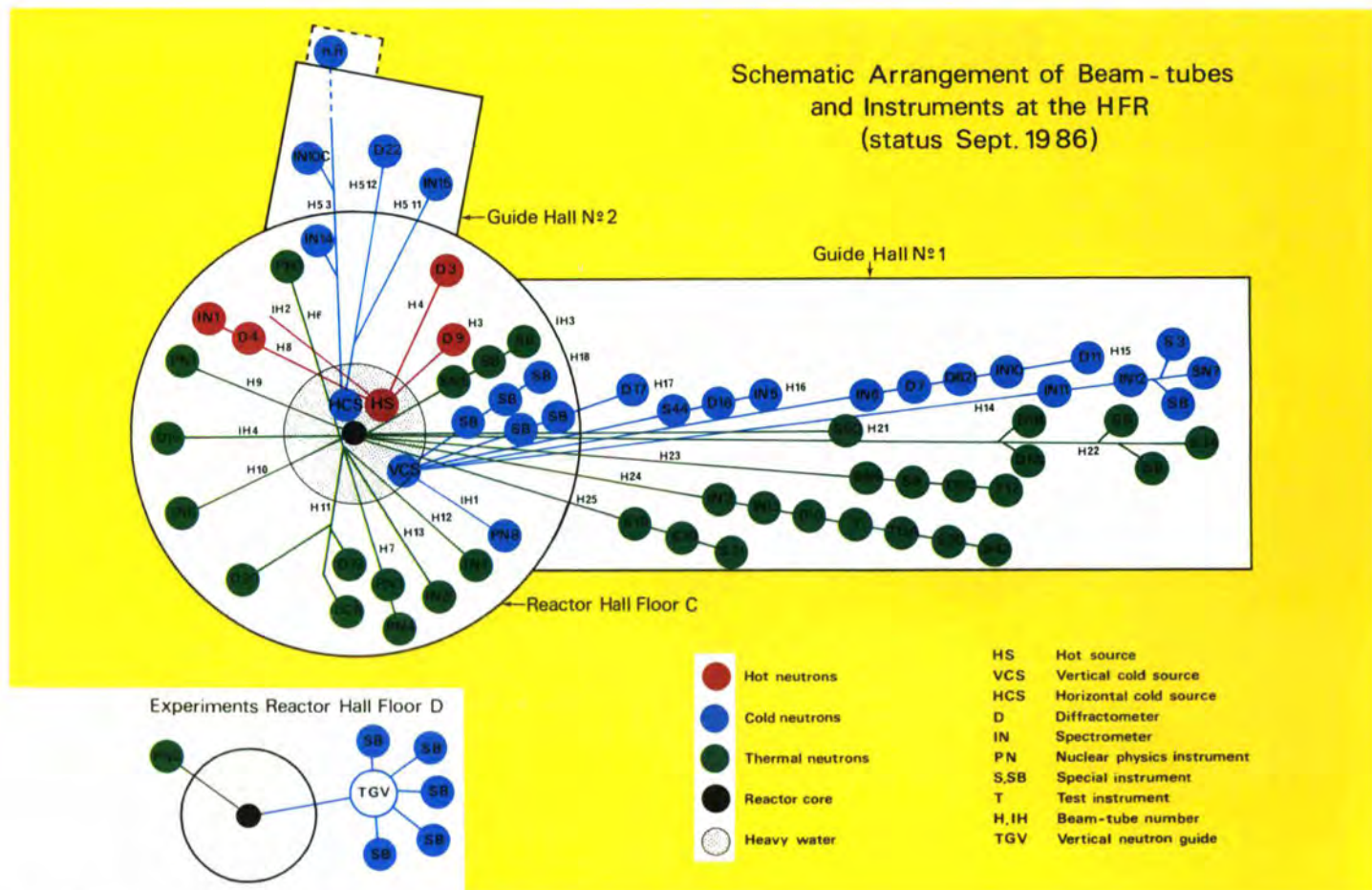
During the first months of 1987 the ILL was caught by the high Tc superconductor fever. These experiments were treated

with high priority and quick access enabled their carrying out outside the normal procedure. The perturbations calmed down in the middle of the year and these experiments - mainly powder diffraction, some single crystal and inelastic neutron scattering work (refer to the sections "Colleges" in this report) - are now scheduled in the framework of the normal procedure.

Altogether 744 experiments were carried out at the ILL after the approval of the two Scientific Councils.

In general, instruments became more modernised and automated to accommodate the experiments which are becoming more elaborate and difficult in terms of sample preparation and sample environment.

Besides English, French, and German one can now hear more and more Spanish: at the instruments and in the corridors of the ILL. About 30 proposals have at least one Spanish participant. In the future there are plans for further countries joining ILL, with the aim to maintain ILL's advanced position and to establish an "international" atmosphere with good scientific collaboration.



Up-dated version of the schematic arrangement of beam tubes and instruments at the HFR (sept. 1986).

# Instrument Statistics - January - December 1987

**Please note:**

The following statistics concern the beam-time allocation of the Scientific Councils of October 1986 and March 1987. Only 5 cycles were run in 1987 due to installations for the new cold source. The instrument allocations refer to a full-power reactor availability of 220 days.

**Not included:**

The statistics concerning the "Special Beam" experiments are not included in the present statistics. They are fully described under "Instrument Operation Department" in this report.

The instrument D3 (polarised neutrons) was rebuilt; no experiment was allocated in 1987.

Instrument Group	Instrument	Beam - Time Request. (Days)	Beam - Time Available (Days)	Fundamental & Nuclear Physics	Structural & Magnetic Excitations	Crystallography & Magnetic Structures	Liquids, Disordered Materials & Metal Physics	Biology	Chemistry, Small & Large Molecules Colloids, Polymers	Comments
Three-axis	IN1 (3-axis)	88	46		37		9			Time sharing on H8 with D4 and IN1 (BeF) Time sharing on H8 with D4 and IN1 (3-axis) 50% availability C.F. special beam allocations
	IN1 (BeF)	53	24				10		14	
	IN3	129	77		28		13		28	
	IN8	312	155		136		19			
	IN12	387	98		118		23	2		
	IN20	345	145		67	78				
Time-of-flight	IN4	264	155		102		35		18	
	IN5	317	166		54		32	10	80	
	IN6	393	163		43		65	13	42	
Back-scattering & spin-echo	IN10	303	159				76	4	75	
	IN11	554	151		44	5	30	12	60	
	IN13	372	165				26		139	
Powder diffraction	D1B	380	170			88	17		65	
	D2B	376	161			146	15			
	D20	76	70			40	11		10	New instrument. Period includes commissioning tests
Single crystal diffraction	D9	486	167			161	6			
	D10	273	171			106	65			
	D15	230	170			170				
	D16	315	176			9		45	122	
	D19	178	161			85		71	5	
	DB21	122	170					122		
Liquids, diffuse scattering	D4	238	110			10	100			Time sharing on H8 with IN1 (3-axis) and IN1 (BeF)
	D7	246	161		8	82	52		14	
Small angle scattering	D11	374	166			8	26	60	72	
	D17	281	173			40	23	8	101	
Nuclear physics	PN1	380	201							Curved crystal spectrometers Gams 1, 2/3 (PN3) and pair spectrometer PN4
	PN2	267	173							
	PN3/4	453	150							
	PN8	428	180							

### PUBLIC RELATIONS

The Scientific Secretary, B. Maier, has produced the first VIDEO-film about the ILL. The duration of the film is 16 minutes and versions in all 3 official ILL languages are available. The film describes, on a popular science level, the task and purpose of the Institut and aims at persons non-initiated in scientific research.

It is our intention to produce, in the forthcoming years, three or four more films on more specific subjects, such as

- Structure of the Institut
- The HFR
- Inelastic Neutron Scattering
- Elastic Neutron and Small Angle Scattering
- Fundamental and Nuclear Physics.

### CHANGE OF STAFF

G. BRIGGS, who has been in charge of the organisation of the experimental programme and the Scientific Council for 14 years, retired on 31 October 1987. The ILL, and in particular SCAPRO, wish to thank him for his excellent work and his commitment to the Institut during that period. His name will be indelibly associated with this Office.

His successor is Dr. Herma BLANK, a mineralogist.



*G. Briggs, who has been in charge of the coordination of the experimental programme in SCAPRO since 1973, retired on 31 October 1987.*



*H. Blank, who has taken over from G. Briggs, is a mineralogist and has been co-responsible for INS for 3 years.*

### WORKSHOPS ORGANIZED BY THE ILL IN 1987

Workshop on "Polymer Motion in Dense Systems",

— ILL Grenoble/KFA Jülich

23-25/9/87

U. Buchenau

B. Maier

A. Rennie

D. Richter

T. Springer

## LIBRARY

### Budget 1987: 717 KF.

The ILL Library continued to maintain collections and to provide users with the information they need. It also purchased literature for ESRF needs.

In 1987,

- 620 books were processed:
  - 180 deposited with ILL departments
  - 120 deposited with ESRF staff,
- 350 subscriptions were handled:
  - 160 deposited with ILL departments
  - 38 deposited with ESRF staff.

An increased number of computerized bibliographic searches and on-line ordering of documents have been carried out.

In close liaison with ILL scientific activity, the library collected:

- 350 publications
- 400 experimental reports.

Discussions are under way on the setting up of a joint ESRF/ILL library.

In 1987, the Library was connected to the central purchasing system "DEAL" and processed 380 purchase orders including 80 for ESRF.

## SAFETY, MEDICAL AND HEALTH PHYSICS GROUP (SPS)

The main function of the units responsible for General Safety, Radiation Protection and the Works Medical Service is to assist ILL staff, guest scientists and employees of outside firms working on the ILL site in the field of safety. For all activities involving a risk, their work makes it possible to define working conditions to ensure that the risks are limited, and to implement appropriate monitoring and inspection facilities.

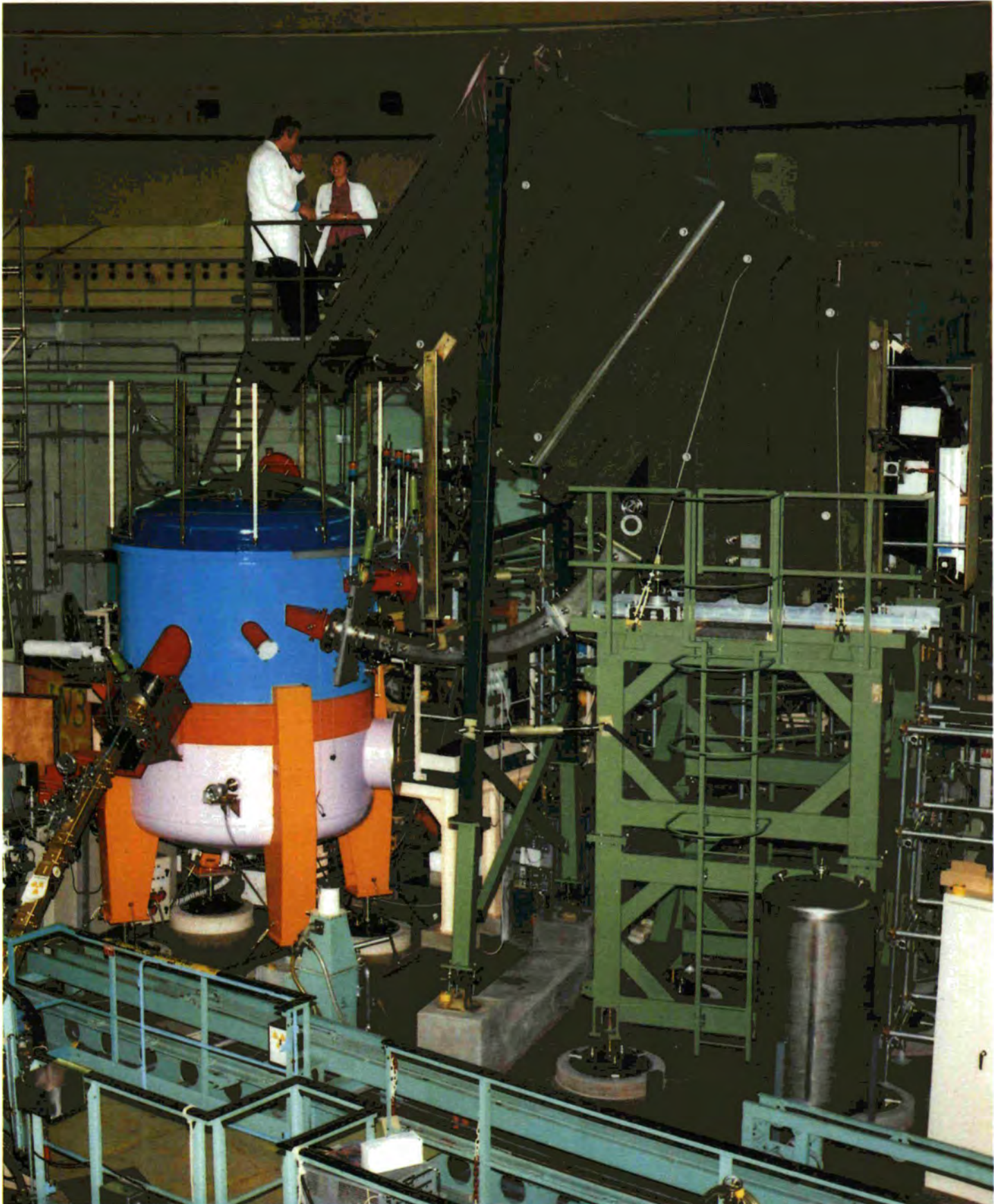
In addition to this day-to-day work, particular attention is paid to the maintenance and improvement of the safety level of the installations. In this connection, the following work was carried out during 1987:

- renovation of certain fire detection installations;
- replacement of measuring instruments in the Health Physics laboratory;
- selection of new equipment for the reactor personnel protection circuit;
- work on the zone for treatment of radioactive waste and storage of reusable radioactive parts
- formalisation of periodical inspections by quality assurance procedures

Among the safety studies or analyses carried out in connection with projects for new installations or for modifications, the following should be mentioned:

- calculations for shielding around the neutron guides (ILL 22) and instruments (D16, S20)
- studies for the neutron-antineutron experiment
- studies for the use of a high activity source (verification of the structure of monochromators by diffractometry)

The units of the Safety, Medical and Health Physics Group also provide technical support for the Committee on Health, Safety and Working Conditions, and for the Internal Safety Commission (CIS).



*The first parts of NESSIE, the neutron gravity spectrometer on "Level D" of the HFR.*

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**INSTRUMENTS AND METHODS DEPARTMENT**

## INTRODUCTION

Le Département Instruments et Méthodes (DIM) est chargé de tout ce qui concerne la réalisation technique des dispositifs expérimentaux et le support technique au développement des méthodes de mesure avec neutrons.

Le département a contribué en 1987 (par la suppression de 3 cases dont 2 dans le secteur de la mécanique) à l'effort général de réduction des effectifs qui devait accompagner la fin du programme de modernisation.

Mais simultanément, les demandes provenant de laboratoires extérieurs en vue de la réalisation par l'ILL de composants d'instruments (détecteurs, monochromateurs) se sont maintenues à un niveau relativement important. C'est pourquoi l'effort a été poursuivi dans la recherche de firmes extérieures susceptibles d'assurer la réalisation technique et le suivi administratif de ces marchés en collaboration avec l'ILL et moyennant le paiement d'une redevance, selon un modèle de contrat en vigueur depuis de nombreuses années déjà dans le domaine de la mécanique.

Parmi les faits techniques marquants il faut citer:

— La définition et le début de réalisation de la structure interne (microfils par dépôt métallique sur verre) du futur détecteur à 1600 cellules de D20.

— A l'occasion d'une commande par HMI Berlin, l'étude d'un grand détecteur (1mx1m) pour diffusion aux petits angles qui devrait servir de prototype à la réalisation du futur détecteur de D22.

— L'installation, les tests, et le début d'exploitation du four presse destiné à la production de cristaux mosaïque de Graphite. Les premiers résultats sont encourageants puisqu'un angle de désorientation de  $0,8^\circ$  a déjà été obtenu sur plus de 30% de la plaque pressée.

— L'installation de l'obturateur de neutrons lié à la source froide horizontale et la pose des guides correspondants dans le hall du réacteur.

— Le début du montage du nouveau 3 axes IN14 qui devrait démarrer à l'été 1988.

— La construction du spectromètre à rétrodiffusion IN10C, du spectromètre Spin Echo IN15 et les premiers travaux en vue de la réalisation de l'instrument de diffusion aux petits angles D22.

Les études de ces nouveaux instruments ont été l'occasion de quelques développements techniques. Citons entre autres:

— Montage d'un guide magnétique de prépolarisation des neutrons en relation avec HMI Berlin et Cilas Alcatel

— Utilisation de poutres en fibres composites articulées sur coussin d'air en n'utilisant que des matériaux entièrement amagnétiques

— Adaptation spéciale d'un multidétecteur aux grandes longueurs d'onde (8-32 Å)

— Collaboration avec Jülich pour adapter un moteur de chopper à une nouvelle électronique de phasage réalisée en VME

Les premières unités électroniques réalisées en VME ont été testées favorablement sur le prototype de IN10C.

Enfin l'année 1987 a permis:

— l'Acquisition en relation avec le département Réacteur d'un poste de travail utilisant le logiciel de CAO (EUCLID (de

Matra Datavision). Ce logiciel sera exploité en commun avec l'ESRF.

— l'Acquisition en relation avec les autres départements techniques, d'un logiciel de calcul des structures mécaniques par la méthode des éléments finis. L'exploitation en 1988 de cet outil devrait permettre de mieux cerner les besoins de l'ILL, importants dans ce domaine (optimisation des pièces de structures dans le respect de la réglementation en matière de sécurité, études des contraintes, etc.)

## INTRODUCTION

The Instruments and Methods Department (DIM) has the entire responsibility for the construction of experimental instruments and the technical support for the development of measuring methods using neutrons.

In 1987 the Department contributed to the overall effort for the reduction of staff following the end of the Modernisation Programme by the elimination of 3 posts, including 2 in Mechanical Construction.

At the same time however, requests from external laboratories for the construction by ILL of instrument components (detectors, monochromators) have remained at a relatively high level. For this reason the effort has been continued to find external firms capable of producing these items and following up the administrative aspects of these contracts in collaboration with ILL on the basis of a licence fee, using a standard contract which has served for a number of years in the mechanical engineering field.

Among the interesting technical projects should be mentioned:

— the definition and start of construction of the internal structure (microwires by metal deposit on glass) of the future 1600 cell detector for D20;

— in connection with an order from the Hahn-Meitner Institut (HMI) Berlin, the design study for a large detector (1m x 1m) for small angle scattering, to serve as a prototype for the construction of the future detector for D22;

— the installation, testing and start of operation of the hot press for producing graphite mosaic crystals. The first results are encouraging, as a misorientation angle of  $0.8^\circ$  has already been obtained over more than 30 % of the pressed plate;

— the installation of the neutron shutter associated with the horizontal cold source and of the corresponding guides in the reactor hall;

— the beginning of the assembly of the new three-axis instrument IN14 due to start operation in summer 1988;

— the construction of the backscattering spectrometer IN10C, the spin-echo spectrometer IN15 and the beginning of the work for the construction of the small angle scattering instrument D22.

The design studies for these new instruments resulted in a number of technical developments. Among these may be mentioned:

— assembly of a magnetic guide for prepolarisation of neutrons in association with HMI Berlin and Cilas Alcatel

— use of articulated composite fibre beams on air cushions, making use only of entirely non-magnetic materials

— special adaptation of a long-wavelength multidetector (8-32 Å)

— collaboration with Jülich to adapt a chopper motor to new VME standard phase electronics.

The first electronic units built to VME standards were tested successfully on the prototype of IN10C.

Other events in 1987 included:

— The purchase jointly with the Reactor department of a workstation using EUCLID CA software (from Matra Datavision). This software will be used in common with ESRF.

— The purchase in conjunction with the other technical departments of software for the calculation of mechanical structures by the finite element method. The use of this system in 1988 should make it possible to define better the ILL's requirements, which are considerable in this field (optimisation of structural parts while respecting the regulations on safety, study of constraints, etc).

## **EINLEITUNG**

Die Abteilung Instrumente und Methoden (DIM) trägt die Verantwortung für die technische Realisierung der Experimentiereinrichtungen sowie die technische Unterstützung bei Entwicklungen von Messmethoden mit Neutronen.

Die Abteilung hat 1987 zur allgemeinen Anstrengung, Stellen nach Beendigung des Modernisierungsprogramms abzubauen, durch den Verzicht auf 3 Planstellen, 2 davon im Konstruktionsbereich, beigetragen.

Gleichzeitig jedoch blieb die Nachfrage von anderen Laboratorien für die Herstellung von Instrument-Komponenten (Detektoren, Monochromatoren) durch das ILL auf einem relativ hohen Niveau. Aus diesem Grund wurde versucht, fähige Industrieunternehmen zu finden, welche die technische Realisierung und die Abwicklung dieser Aufträge in Zusammenarbeit mit dem ILL im Rahmen eines Lizenzvertrages sicherstellen. Ein seit einigen Jahren gültiger Lizenzvertrag im Konstruktionsbereich dient hierfür als Vorbild.

Folgende technische Entwicklungen sollten erwähnt werden :

— Spezifikation und Herstellungsbeginn der inneren Struktur (Mikroelektroden durch Metallbeschichtung von Glas) des zukünftigen Detektors mit 1600 Zellen für D20.

— Entwurf eines grossen Detektors (1m x 1m) für Kleinwinkelstreuung, der als Prototyp für die Realisierung des zukünftigen Detektors für D22 dienen wird (Bestellung des HMI, Berlin).

— Aufbau, Tests und Betriebsbeginn des Pressofens zur Herstellung von Mosaik-Graphitkristallen. Die ersten Ergebnisse sind ermutigend, da ein schon bei über 30 % der gepressten Platten ein Mosaikwinkel von 0.8° erreicht wurde.

— Aufbau des Neutronenverschlussventils im Zusammenhang mit der Horizontalen Kalten Quelle und Montage der entsprechenden Leiter.-Anfang der Aufbauarbeiten des neuen Dreiaxieninstruments IN14, das im Sommer 1988 anlaufen soll.

— Der Bau des Rückstreuungsspektrometers IN10C, des Spinechospektrometers IN15 und erste Arbeiten im Hinblick auf die Realisierung des Kleinwinkelstreuungsinstruments D22.

Die Studien für diese neuen Instrumente gaben Gelegenheit, einige technische Entwicklungen voranzutreiben. Unter anderem können folgende genannt werden:

— Einbau eines magnetischen Leiters für die Vorpolarisierung von Neutronen in Verbindung mit dem HMI, Berlin und Cilas Alcatel.

— Verwendung von Trägern aus Verbundwerkstoffen, die auf Luftkissen verfahrbar sind und für die vollkommen unmagnetische Werkstoffe benutzt wurden.

— Besondere Anpassung eines Multidetektors an grosse Wellenlängen (8-32 Å).

— Zusammenarbeit mit Jülich, um einen Chopper-Motor an eine neue in VME realisierte elektronische Phasenregulierung anzupassen.

Die auf dem Prototypen von IN10 erzielten Testergebnisse der ersten in VME realisierten Elektronikeinheiten waren positiv.

Schliesslich wurden im Jahr 1987 folgende Ergebnisse erzielt:

— Erwerb zusammen mit der Reaktorabteilung eines Terminals für computerunterstütztes Zeichnen (Software EUCLID von Matra Division). Diese Software wird gemeinsam mit ESRF benutzt werden.

— Erwerb in Verbindung mit den anderen technischen Abteilungen einer Software für die Berechnung der mechanischen Strukturen durch die Methode der Fertigteile. Die Benutzung dieses Hilfsmittels sollte im Jahre 1988 erlauben, den in diesem Bereich bedeutenden Bedarf, wie z.B. Optimierung der Strukturteile unter Beachtung der Bestimmungen hinsichtlich Sicherheit (Festigkeitsuntersuchungen) des ILL besser nachzukommen.

## MECHANICAL CONSTRUCTION

This group includes three separate sectors:

### 1. Design Studies

In 1987 this sector contributed to the completion of the IN14 project, 60 % of which had already been built, and to the successful conclusion of the powder diffractometer D20. Design studies carried out in association with the Reactor Department permitted the shutter for the new Cold Source to be completed under quality assurance conditions. The studies to define the spectrometers IN15 and IN10C necessitated the use of new materials, and for certain parts required examining technological solutions in fields which had never been used at ILL. This was the case for the use of composite materials for instrument structures (IN15) or for chopper rotors and selectors using carbon Kevlar composites containing neutron absorbent products. Finally it was possible to order the detector tube following design studies for the future diffractometer D22, largely based on the recent reconstruction of D11.

### 2. The Workshop Sector

was in great demand for mechanical elements for the new Cold Source, for equipment for new instruments such as IN20, IN14, D20, D3. This sector acquired very good skills in connection with technical developments associated with the detectors, focussing devices, monochromators and high speed choppers of composite materials (650 m/sec peripheral velocity).

All these projects did not prevent immediate services to the users of the operational instruments, to which the workshop devoted more than 30% of its activity.

### 3. The Assembly Sector

has carried out major work on D2B, D20, IN20, D11, IN1B, and the reconstruction of the diffractometer D3, transferred during the year from the thermal beam H5 to the hot beam H4. The Horizontal Cold Source beam shutter was also commissioned, and at the beginning of the installation of IN14 the daily assistance to the instruments in operation represented 40 % of the activity of the sector. The recent installation in the metrology laboratory of a computerised test bench should also be mentioned, which has already permitted the adjustment and the inspection of new direct coding mechanical modules giving accuracy between  $\pm 0.001^\circ$  and  $\pm 0.0025^\circ$ .

## MULTIDETECTOR GROUP

The major items of work included:

1. Design and construction of the detector for the new instru-

- ment IN15
2. Completion of work on the former D11 detector and adaptation as a spare for the present instrument
3. Tests and adjustments of detectors for D9 and D15
4. Continuation of tests on the low resolution 16000 cell  $^3\text{He}$  detector for DB21
5. Development of delay line electronics for the above detectors
6. Equipment of instrument IN5 with 40 arrays of detectors
7. Assistance with the reinstallation of experiments S18 and S21
8. Rewiring of instrument IN3
9. Miscellaneous work on instruments D5, D20, D2B, D11

## Detector Laboratory

— Commissioning of the test bench with mobile  $\alpha$  source, simulation of problems observed on the detectors for D9 and D15

— Equipment for differential filling on the  $^3\text{He}$  bench with a view to the filling of the HMI detector

## Others

Preparation of a poster for the London conference

— Development on an IBM/PC of an acquisition system for tests on "canal technique"

— Reconstruction of 'canal technique' No 2, recently equipped with a precise system for optical control of the incident beam

In 1987 we have done work for several external laboratories:

— Laboratoire Léon Brillouin: electronic equipment of 2 multidetectors with 400 and 640 cells

— HMI-Berlin:

Electronic equipment of 400 detectors for a time-of-flight instrument

Design studies for a large 4000 cell multidetector (these studies can be completely re-used for the future instrument D22)

— Saclay: Construction of a multidetector identical to that developed for DB21

We have taken advantage of this work to rebuild and modernise the 'ILL standard' electronics dating from 1975.

## ELECTRONICS GROUP

IN3B: Conversion to CAMAC. development of individualised electronics for PRECILEC encoders (synchronous type) and commissioning (with PDP11)

D1B: Conversion to CAMAC and commissioning (with PDP11)

IN10A: First acquisition of VME data and transfer to DEC10 (for comparison with the data acquired in parallel via CAMAC)

Positive result on a rate of 1 k events (1 kHz), the VME treats 10 % more data than the previous electronics (CAMAC + PDP11)

IN2B: Preparation of equipment for changing the computer plus electronics (end 1987 - beginning of 1988)

## VME Development

### 1) Conversion of the control and acquisition electronics to VME under a system

- a) Manual mode - MACINTOSH
- b) Programmed mode - OS9 system

### 2) Development of VME cards

- a) Channel Card for data acquisition (multidetector or individual detectors)
- b) Time Generator to generate additional parameters in association with a DOPPLER machine (IN10)
- c) Time Generator to generate additional parameters in association with a chopper
- d) Control of a synchronous or hysteresis motor for speed and phase (for machines analysing time of flight) - "chopper control system"
- e) Single axis control of a DC or step motor plus absolute or incremental angle encoder.

#### Laboratory:

Development of a centralised microprocessor development system with SESAD work stations.

## MULTILAYER LABORATORY

(O. Schärpf, W. Kaiser, W. Graf)

Both electron beam evaporators have been operated with an availability rate of better than 90%. Until October 1987 more than 40 m<sup>2</sup> of Titanium-Cobalt supermirrors have been produced during 220 evaporation operations. Eleven more operations were necessary for coating neutron guide elements with Titanium-Nickel supermirrors. The total represents a value of 2.8 MF.

The supermirrors are used to replace single-face coated polarizers on the diffuse scattering instrument D7 by double-face coated polarizers, to equip the 3-axis spectrometer IN12 for polarised neutron measurements and to satisfy orders from the Laboratoire Léon Brillouin, Orme les Merisiers, France and McMaster University, Chalk River, Canada.

The neutron guide elements are assembled in the H511 neutron guide on the horizontal cold source (beam splitter for the new spin echo spectrometer IN15) and neutron guides at KfA Jülich and GKSS Geesthacht, Germany.

A new optical bench equipped with a laser has been installed permitting the curving of the bender polarizers with high precision before the final tests with neutrons on the S3 set-up. The latter was used to test the performance of the recently delivered sputtering equipment from the HMI Berlin, and to measure the first good quality supermirror produced by sputtering techniques from an industrial manufacturer, using a method developed by J.B. Hayter, Oak Ridge National Lab., USA.

New developments at ILL are continuing to improve supermirror characteristics. First results are encouraging.

## MONOCHROMATORS

(A. Freund, B. Hamelin)

D13A : Neutron double crystal diffractometer on thermal guide H22

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 has been moved to a new position on H24 waiting for a decision on a polyvalent test site equipped with a multiple monochromator device including polarizing crystals. This test station will accommodate various facilities such as T12, D13A, Cryopad.

D13C has been working very reliably all over the year. Many test experiments were carried out, for instance on surface diffraction below the critical angle of total reflection and on the stability of pyrolytic graphite under strong acceleration. Crystal characterization, orientation and assembling were performed for the preparation of high efficient monochromators. Besides its use by ILL staff also in 1987, this instrument has shown to be very attractive to external users which made scheduling not always easy, but created a very lively atmosphere.

Li2A and B have been used normally for X-ray orientation and test of single crystals. No major modifications were made.

Li3A-F, the five gamma-ray diffractometers, had to work without the cold source because of the shut-down of the Siloë reactor, which, as we now know, is only temporary. However, the long-lived cesium source permitted to pursue crystal testing before, during and after plastic deformation, during crystal growth and all the usual work needed for monochromator production. Besides this, several experiments in solid state physics were carried out which require the specific advantages of high energy photons such as low absorption and small extinction. Typical examples are studies of the perfection of Co<sub>8</sub>Pt during annealing and of ZnCo single crystals during growth, investigations on the relation between the mechanical strength of Ni-based superalloys and their mosaic structure, and testing of the crystalline quality of various samples, for instance CoF<sub>2</sub>, SiGe, Au<sub>0.8</sub>, Ni<sub>0.2</sub>, ZrNb<sub>16</sub> and Al<sub>2</sub>O<sub>3</sub>.

In order to develop new techniques of crystal characterization, which is also one of the tasks of the Monochromator Group, the possibility to use high voltage X-ray generators for the production of radiation between 60 keV and 400 keV has been studied. First results of intensity measurements on an industrial apparatus showed that fluxes, at least similar to those delivered by nuclear sources can be obtained. Therefore, provided the development of suitable optics and instrumentation, this X-ray generator would be a very useful complementary source for the kind of experimental work performed by the Monochromator Group.

### Monochromator Production and Development

The new IN8 monochromator (Ge111, 160 x 160 mm<sup>2</sup>) with tunable focussing was installed. For D20, a first graphite monochromator was manufactured, which will be replaced once better graphite will be available from the ILL's own production (see below). However, even with the presently available crystals carefully selected from material supplied by Carbone Lorraine, the flux at the sample is high enough for many experiments and detector testing at D20. The Ge511 monochromator for the same instrument and the Cu220 monochromator for IN20 are nearly finished now and will be installed in the beginning of 1988.

Some time was spent on crystal realignments, which appear to be necessary after several years of use or after some unfortunate manipulations. This was required for the monochromators of IN3, IN13, IN20, IN12 and some others. It is evident that the phase of production for instruments constructed within the modernisation programme is at present coming to an overlap with increasing research and development work on novel materials, designs and devices. Efforts are needed to maintain ILL's competence and expertise at a high level and quite generally to still increase the efficiency of an experimental technique which is much more source limited than others, in particular X-ray scattering using synchrotron radiation!

In this context, it was very encouraging that five of the seven partners of the Be project decided to continue supporting the activities at the beryllium laboratory of the Max-Planck-Institut für Metallforschung in Stuttgart. The contract was extended for another three years beginning April 1987 and the new membership of the Technische Hochschule Aachen permitted to solve problems with staff contracts. The new triple ellipsoid furnace started in January 1987 and has worked since without major problems, while the double ellipsoid furnace had to be overhauled. After initial delays of the production programme, the rate of crystal growth is now according to schedule. The crystal quality is reproducibly high if the crystal diameter stays below about 15 mm, but there is a very strong decrease in the success rate for thicker crystals. Therefore, a method of automated crystal orientation and assembling is under study to decrease the time needed for preparing the final monochromators. The same device is required for composing a thick crystal of fairly large mosaic spread using several thin lamellae of small mosaic spread produced by spark machining, which further was shown to give a very uniform mosaic structure. Detailed reflectivity studies of such composite elements are under way and it is worth noting that they correspond to concepts of lamellar systems which were proposed at the very beginning of ILL's existence.

Despite the absence of in-beam control by gamma-ray diffraction, first success was achieved at the pressing of pyrocarbon plates to produce highly orientated pyrolytic graphite. At present, circular plates 10 mm thick and 120 mm in diameter can be obtained with a mosaic spread of about 0.8° and locally even less. Financial support by the National Bureau of Standards was greatly appreciated and will be used to purchase a HF generator so that the whole facility can be transferred from the CNRS to the ILL. The Cs source and the equipment for real-time control of sample quality as a function of pressure, time and temperature will be installed soon in 1988. This will

allow for both the finding of the optimum parameters and the correcting of variations in the quality of the material at the beginning and during the pressing process. Thus, it should be possible in the near future to reach the physical limit of HOPG quality.

The SiGe project on the production of crystals with slightly variable lattice constant or a gradient in the lattice constant (in conjunction with C. Holm, Stammham) has been progressing. Much time was spent on improving the properties of the starting material, which lead to the preparation of uniform Si<sub>0.9</sub>Ge<sub>0.1</sub> mixed crystals without the optically visible defects which had been observed before. A more favourable temperature distribution inside the furnaces and an improved heating efficiency now permit the optimization of the growth conditions according to the desired orientation of the lattice planes and to use four transport systems simultaneously. The transition from the development to the production phase is expected for next year.

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**REACTOR OPERATION DEPARTMENT**

## INTRODUCTION

En 1987, les cycles de fonctionnement du Réacteur ont été programmés en fonction des périodes d'arrêts imposées par les travaux liés à la mise en place de la nouvelle source froide et à l'implantation des faisceaux de neutrons qui en sont issus vers les nouveaux halls expérimentaux.

Dans ce contexte, cinq cycles complets de fonctionnement ont, néanmoins, été réalisés.

## INTRODUCTION

In 1987 the Reactor operating cycles were scheduled as a function of the shut-downs necessitated by the work in connection with the installation of the new cold source and of the neutron beams from this source to the new experiment halls.

It was, however, still possible to complete five operating cycles.

## EINLEITUNG

Die Betriebszyklen des Jahres 1987 waren weitgehend von der Horizontalen Kalten Quelle bestimmt. Dabei waren vor allem die Arbeiten für den Einbau in den Reaktor und für die Einrichtung der Neutronenleiter für die neuen Experimentierhallen entscheidend.

Trotz dieser Arbeiten sind fünf vollständige Betriebszyklen erreicht worden.

# REACTOR OPERATION 1987

### CYCLE 1/87

Operation from 6 January to 19 February. The scheduled dates were respected and the cycle was completed without incident.

### CYCLE 2/87

Operation from 17 March to 30 April. The scheduled dates were respected and the cycle was completed without incident.

### CYCLE 3/87

The cycle was originally scheduled to run from 12 May to 25 June, and was extended until 29 June to compensate for a shut-down for work on the control rod mechanism.

### CYCLE 4/87

Operation from 7 July to 20 August. The scheduled dates were respected and the cycle was completed without incident.

### CYCLE 5/87

Operation from 29 September to 12 November. The scheduled dates were respected and the cycle was completed without incident.

## DATA FOR 1987

Number of days originally scheduled	220
Actual number of days of operation	220.2
Number of equivalent days of full power	211.55
Actual operating time as proportion of year	60 %
Actual operating time in relation to time scheduled	100 %
Number of fuel elements used	5
Number of fuel elements despatched for reprocessing	6
Number of new fuel elements received	5
Number of unscheduled shut-downs	1
including:	
brief shut-downs	0
shut-downs with Xenon poisoning	1

## WORK OF THE VARIOUS GROUPS

In addition to the usual maintenance work on all the installations during the normal shut-downs, certain exceptional operations were carried out in 1987, in particular:

## PREPARATION, INSTALLATION AND TESTS ON THE NEW HORIZONTAL COLD SOURCE

The three long shut-downs scheduled in 1987 (19/2 to 17/3; 20/8 to 29/9; 12/11 to end 1987) were for the installation of the Horizontal Cold Source in beam tube H5 and of the neutron guides leading from this beam tube to the new neutron guide hall ILL 22.

During the first half of 1987, the various components of the Horizontal Cold Source were subjected to acceptance tests, assembled and tested separately; the cryogenic unit was then tested after being connected to a model outside the Reactor. As all these preliminary operations were satisfactory, the final installation and connection to the Reactor installations began on 12 November and finally started operation, with the Reactor running.

Its operation is in accordance with expectations. It may be noted that the nuclear power received by the new source is approximately 3kW (calculated figure 2.5 kW). This power, which is additional to that received by the vertical cold source (6 kW), remains compatible with the available power of the helium refrigerator (11 to 12 kW).

The first results for the neutron flux from the new source are given elsewhere. They largely confirm the calculated values in the region  $\lambda > 3\text{\AA}$ .

## **INSTALLATION OF THE NEW HELIUM COMPRESSOR OF THE REFRIGERATOR FOR THE COLD SOURCES**

This screw compressor is intended to replace the piston compressors which have been in use since the start of the ILL. It was installed and tested during the third quarter of 1987.

Its performance is in accordance with the specification. The control cabinet for the integration of this compressor in the refrigerator was ordered at the end of December 1987 and will be installed during the next long shut-down (mid-1988).



*Installation of the neutron guides on the second cold source*

## **SHUT-DOWN FROM 19/2 TO 17/3/87**

Construction of the primary 'casemate' at the H5 beam outlet. Piercing of the two shells of the Reactor building and installation of square sleeves to permit passage of the neutron guides from H5.

## **SHUT-DOWN FROM 20/8 TO 29/9/87**

Connection of the deuterium and helium circuits to the Horizontal Cold Source model outside the Reactor, and cryogenic tests.



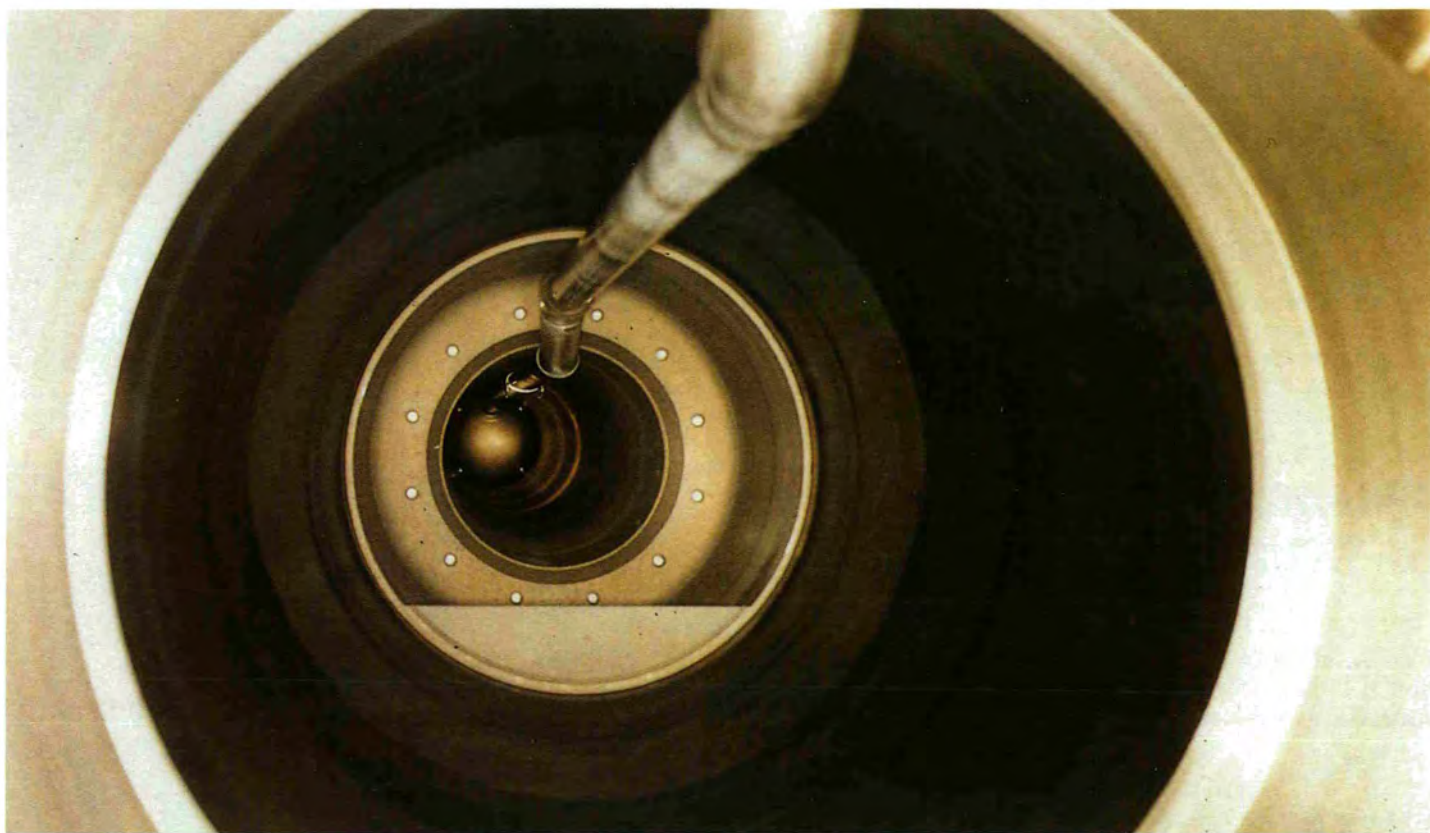
*Shunt of neutron guides for the second cold source*

## **SHUT-DOWN FROM 12/11 TO END DECEMBER 1987**

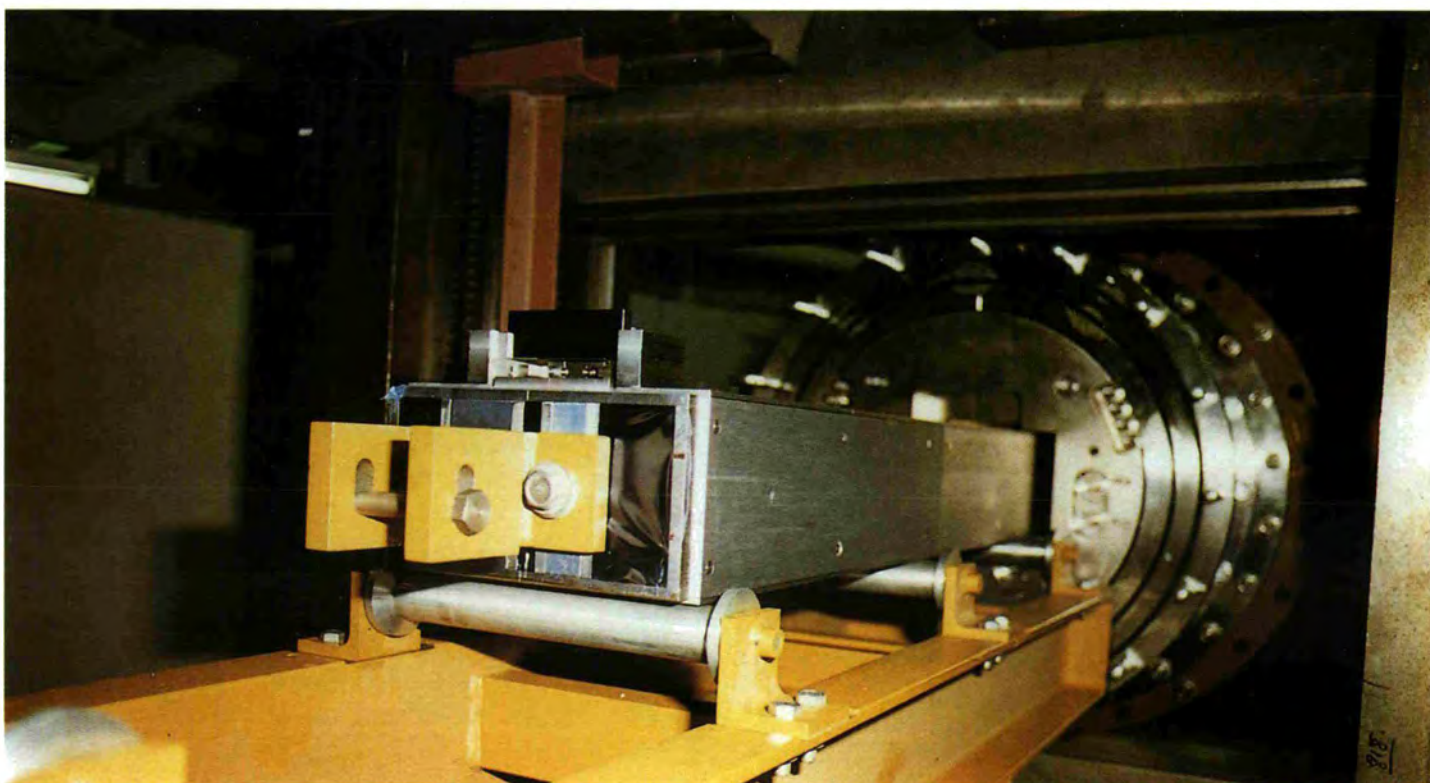
Installation of the new H5 beam tube thimble.  
Installation and associated testing of the complete Horizontal Cold Source.  
Installation of the neutron guides from H5.

## **LEAK-PROOFING OF THE CONCRETE SHELL OF THE REACTOR BUILDING**

Following the work in 1986 on the exterior of the building, in 1987 epoxy resins were injected into the hairline cracks found inside the shell at the level of the ring between the cylindrical wall and the dome of the Reactor.



*Inside view of the 2nd Cold Source liner with the liquid D<sub>2</sub> moderator cell at the bottom in the middle. This cell is connected to concentric pipes for the filling and overflow of the deuterium.*



*The photograph shows the rear flanges of the beam port H5. The block of metallic guides for the extraction of three neutron beams is shown in the middle of the plug.*

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**COMPUTING DEPARTMENT**

## INTRODUCTION

The year has seen some notable events in the mainstream activities of the Department, principally the decision to replace the ageing DEC-10 Central Computer by a VAX 8700, expansion of network services, and continuing replacement of the older instrument control computers, albeit not quite as quickly as would be desirable.

Standardisation of equipment is thus being slowly but steadily achieved. The Ethernet network supposes that most computers linked to it can support DECNET protocols. The Central Computer Facility will in future operate with a homogeneous VAX 8650/VAX 8700 cluster. And new instrument computer systems will, as a general rule, be either a microVAX II (if the work-load justifies it) or a PDP 11/73.

On the other hand, one observes a proliferation of personal computers, in respect of which the Department's role is still being worked out. Some are used as intelligent terminals on established instrument systems. But many are being used for totally new activities. In no way are they taking any load off or diminishing the need for the larger shared machines.

Requests to provide more and better facilities in the office automation area are causing some problems, since the available resources are clearly unequal to all the demands. Members of the Department played a key role in the installation of a new package for the Finance and Purchasing Service.

Collaboration with the ESRF has continued on a mutually satisfactory basis. The ESRF is part owner of the VAX 8650, and thereby has rights to use one-third of its capacity. The Ethernet network has been extended to pass through the ESRF building. We provide other cabling support for that building, and also look after their telephone system, which is now independent of the ILL system. Their special requirements in respect of salaries and accountancy calculations have also been taken in-hand.

## INTRODUCTION

Cette année quelques événements importants se sont réalisés dans les principaux domaines d'activité du Département, parmi lesquels on notera principalement la décision de remplacer l'ancien ordinateur central DEC-10 par un VAX 8700, l'extension des services réseaux et le remplacement progressif, mais pas aussi rapide que souhaitable, des plus anciens calculateurs de contrôle d'instruments.

La standardisation de notre équipement est, de la sorte, en train de se réaliser lentement et de façon continue. Le réseau ETHERNET laisse supposer que la plupart des calculateurs qui lui sont connectés puissent supporter les protocoles DECNET. Les systèmes du Centre de Calcul fonctionneront dans le futur dans un cluster homogène avec les VAX 8650/VAX 8700. Et les nouveaux calculateurs d'instruments seront, en règle générale, soit un microVAX II (si la charge de travail le justifie), soit un PDP 11/73.

D'autre part, on observe une prolifération des ordinateurs personnels pour lesquels le rôle du Département est encore à l'étude. Certains sont utilisés comme des terminaux intelligents sur les systèmes d'instruments opérationnels. Par contre, beaucoup s'utilisent pour des activités totalement nouvelles. En aucun cas ils ne réduisent la charge de travail sur les plus grandes machines multi-utilisateurs et donc ne diminuent en rien leur besoin.

Les demandes en vue d'apporter un meilleur service dans le domaine de la bureautique créent quelques problèmes, dans la mesure où les ressources disponibles sont insuffisantes pour satisfaire toutes ces demandes. Les membres du Département ont joué un rôle clé dans la mise en place d'un nouveau logiciel pour le Service Financier et Achats.

La collaboration avec l'ESRF s'est poursuivie sur la base de la satisfaction mutuelle. L'ESRF est en partie propriétaire du VAX 8650 et, de ce fait, a le droit d'utiliser un tiers de la capacité de cette machine. Le réseau ETHERNET a été étendu jusqu'au bâtiment ESRF. Nous leur fournissons également une assistance pour le câblage de leur bâtiment et pour leur système téléphonique qui est indépendant de celui de l'ILL. Leurs demandes particulières en ce qui concerne les salaires et les calculs en comptabilité ont été également pris en charge par le Département.

## EINLEITUNG

In diesem Jahr waren einige bemerkenswerte Ereignisse in den Haupttätigkeitsbereichen der Abteilung zu verzeichnen. In erster Linie kann die Entscheidung genannt werden, den alternativen DEC-10 Zentralrechner durch einen VAX 8700 zu ersetzen sowie die Ausdehnung der Netzwerk-Dienste und den Ersatz weiterer älterer Instrument-Kontrollrechner, obgleich dies nicht so schnell wie gewünscht voranging.

Langsam aber sicher wird auf diese Art die Standardisierung der Ausstattung erreicht. Das Ethernet-Netzwerk setzt voraus, dass die meisten angekoppelten Rechner DECNET Protokolle zulassen. Der Zentralrechner wird zukünftig mit einer homogenen VAX 8650/VAX 8700-Rechnerverkopplung betrieben werden. Die neuen Instrumentrechnersysteme werden in der Regel entweder ein MikroVAX II (wenn die Arbeitslast dies rechtfertigt) oder ein PDP 11/73 sein.

Andererseits ist eine starke Erhöhung bei den PCs zu beobachten, für die die Rolle der Abteilung noch zu klären ist. Einige davon werden als intelligente Terminals auf etablierten Instrumentsystemen benutzt. Viele andere hingegen werden für vollkommen neue Tätigkeiten verwendet. In keiner Weise bedeutet dies eine Entlastung oder eine Bedarfsverringerung von grösseren in "time-sharing mode" betriebenen Rechnern.

Die Nachfrage für mehr und bessere Geräte im Büroautomatisierungsbereich führte zu einigen Problemen, da die verfügbaren Mittel eindeutig nicht ausreichen, alle Anfragen zu befriedigen. Mitarbeiter der Abteilung spielten die Schlüsselrolle bei der Installierung des neuen Programmpaketes für die Einkaufs- und Finanzabteilung.

Die Zusammenarbeit mit ESRF wurde auf einer gegenseitig zufriedenstellenden Basis fortgesetzt. Die ESRF besitzt einen Anteil des VAX 8650 und ist dadurch berechtigt, ein Drittel der Rechnerkapazität in Anspruch zu nehmen. Das Ethernet-Netz wurde erweitert und durch das ESRF-Gebäude durchgeführt. Für dieses Gebäude wurden weitere Verkabelungen von uns bereitgestellt. Ausserdem kümmern wir uns um das Telefonsystem der ESRF, das nun unabhängig vom ILL-System arbeitet. Ihre besonderen Anforderungen hinsichtlich Gehälter und Finanzbuchhaltung wurden ebenfalls von uns berücksichtigt.

## **AUTOMATION AND DATA SERVICE (SAD)**

A number of dominant themes may be noted.

Firstly, the arrival of networks as an essential part of the facilities offered by the Service. Whereas the Ethernet project was until fairly recently seen as of strictly limited interest, we are now facing up to the need to provide connections throughout the Site.

Secondly, scientists are increasingly expecting to have high-quality graphics terminals on the instrument computer. The ability to instantly display current data is seen to be of value in exploiting to a maximum available beam time.

Thirdly, it is becoming evident that all the PDP 11/34 computers must be replaced as quickly as possible. These machines, which are mainly between 8 and 10 years old, are beginning to show serious signs of wear, cannot accommodate enough memory for future needs, and have outdated peripherals. During 1987, six of these systems were either replaced or firmly scheduled for replacement early in 1988. But there still remain eight in service, many of them on high-profile instruments.

### **SOFTWARE A (CRYSTALLOGRAPHIC INSTRUMENTS)**

**(A. Barthélemy)**

This Group is responsible for software for the instruments D1A, D1B, D2B, D3B, D4, D9, D10, D15, D16, D19, D20, DB21, LI5, LI8, S18.

It also has global responsibility for the Ethernet network, and for DEC-licensed software on VAX-range machines.

The progressive modernisation of instrument computers has continued with all possible speed.

The first MicroVAXII to be installed on an instrument was for D9. Advantage was taken of this change to completely rewrite the program package, and to introduce a procedure for data compression, to better use the available disc space. This latter technique has also been applied on D19.

PDP 11/73s have been installed on D1A and D3B, and a PDP 11/24 on D1B.

Considerable efforts have continued to be put into instrument control (notably temperature control), data collection and data treatment.

In response to pressure from scientists working on the powder diffractometers a project was put in hand (with the help of external mathematicians) to apply better statistical techniques to the fitting of spectra. This has resulted in the production of a now widely-used program, ABFFit, which is available in VAX and MacIntosh versions.

The Group has been active in the area of VME, seen as a successor to CAMAC for electronics on instruments. It supports the microVAX used for these studies, and detached a programmer to the Project when this became independent of the Service.

In the field of development, the Group has acquired a VAX Station 2000 and is studying, especially, the graphics possibilities, in collaboration with the Graphics Group of the Central Computer Service.

Work in the Ethernet area is expanding rapidly. It has, almost over-night been transformed from a study project to a site-wide service, with the whole of the ILL site due to be cabled up as quickly as funds will allow. At the same time, the range of devices being connected is increasing. DECservers are being introduced, not only to link-in terminals, but also to enable printers to be shared. There is also a demand for the connection of non-DEC equipment, notably IBM-PCs (or equivalent) and MacIntoshes.

At the same time, the longer-established JUDAS network, for transfer of instrument data to the central computers, is continuing to function reliably.

The Group undertakes the distribution to the whole of the Department of DEC-supplied VAX software. This is intended to ensure that up-to-date and mutually compatible versions are in use throughout ILL, clearly desirable as more and more computers are communicating with each other over networks.

### **SOFTWARE B (OTHER NEUTRON SCATTERING INSTRUMENTS)**

**(R. Ghosh)**

Software B supports the instruments in the Three-Axis (IN1, IN1BeF, IN3, IN8, IN12, IN14, IN20) and Vercors (IN4, IN5, IN6, IN10, IN11, IN13, D7, D11, D17) Groups.

Operating systems and libraries are kept up to date, together with the standard control software for data acquisition and data transfer programs. One member of the Group has been temporary seconded to lead the VME implementation project on IN10.

In the Three-Axis area the long sought after goal of complete unification of computer hardware has finally been reached, using the PDP 11/73. Three such machines were acquired during the year, one to replace the SOLAR 16/40 on IN12 (with new electronics this will come into operation in 1988) one to replace the PDP 11/24 on IN20 (a machine which now sees service on D1B) and one for the new instrument IN14. Also in this area, considerable work has been needed on IN3, following modifications to its electronics.

In the Vercors area, a considerable effort was put into modernising IN13. This is an instrument which, because of staff shortages, had received insufficient support in recent years, and was, as a result, falling below acceptable standards of performance. A PDP 11/73 was acquired and two members of the Group were assigned to completely rewrite the control program. The new version was installed ahead of schedule, to the complete satisfaction of the instrument scientists.

Some small but significant improvements have been made to D11 and D17. Automatic sample temperature control is available, and the data collected on the multidetector can be displayed in colour, using a low cost personal computer linked to the VAX 11/730. This is used for detector maintenance as well as by scientists.

The area now seen to merit top priority is time-of-flight (IN4, IN5, IN6). Their computers are all now ten years old and have insufficient capacity to permit improvements. It is intended to start replacing these systems in 1988.

One member of the Group provides support for MacIntosh software. This has become the most popular type of personal computer in use at ILL, not only amongst scientists, but also in the technical services. Whilst, by their very nature, they are used autonomously, the Department is trying to ensure that there is some coordination, and not too much dispersion and duplication of effort.

### **SOFTWARE C (NUCLEAR PHYSICS AND SPECIAL INSTRUMENTS)**

**(Ph. Ledebt)**

In the Nuclear Physics area the Group supports the instruments PN2 (BILL), PN3 (GAMS 1,2,3,4), PN4, PN6 (OSTIS).

It is also intended that the Group will take over responsibility for PN1 (Lohengrin), which is due to have its computer replaced in 1988.

The principal Special Instruments supported are S3, S20, S34, S53, SN7, NESSIE and the TGV assembly.

### **MANAGEMENT INFORMATION SYSTEMS AND OFFICE AUTOMATION**

**(M. Grevaz)**

The principal activity of the year was assisting with the introduction of a new unified software package for the Finance and Purchasing Service, running on in-house computers. It had been hoped that following the initial installation phase the MIS Group's role in this would rapidly drop back to that of maintaining the system and providing advice. Unfortunately, staff changes and sickness in the Finance Service have delayed their taking over the product, and the MIS Group has been obliged to continue to provide support on a daily basis. This has seriously perturbed the Group's ability to work in other areas.

Such other activities as were possible have included the introduction of an additional microVAX II to resolve a problem of overload in the secretaries' text-handling service, and the connection of the Group's 3 VAX computers to the general Ethernet network. A TELEX facility is now incorporated in the principal text-handling service.

The Group has worked with prospective users of a Computer Aided Design system, leading to the choice (in line with an ESRF decision) of the MATRA EUCLIDE package running on a VAX/GPX.

It should be noted that the ESRF are customers of many of the packages supported by the Group, and their special needs

have been taken into account, notably in the areas of salaries, accounting and travel claims.

The list of work waiting to be undertaken has grown relentlessly during the year. It is evident that there is a serious gap between the expectations of the various ILL Services of being provided with more and better automated office systems, and the resources available for implementing them. This matter will have to be given serious attention in 1988.

### **COMPUTER MAINTENANCE**

**(J.J. Tschofen)**

The two members of this Group can only, themselves, undertake maintenance of a small fraction of the equipment installed. For the rest, a number of options are available: a contractual guarantee of intervention at ILL, replacement of faulty units, repair in the contractors' own workshops etc. Every effort is made to adopt the most cost-effective solution for each range of equipment.

There are a number of other activities for which in-house staff offer the only realistic solution. Considerable savings can be made by shuffling modules and peripherals between different systems, and even switching computer systems between instruments, but this requires a good knowledge of the complete set of equipment available. The Group maintains full records of the configurations of all systems on site.

### **TELECOMMUNICATIONS**

**(R. Pouget)**

The ILL telephone system is fairly stable at the present time, but the Group undertook the installation of the independent telephone switchboard for the ESRF, and continues to provide support.

The continuing expansion of the various computer networks brings a permanent requirement for cabling assistance. Whilst the cabling work itself is contracted out, planning and supervision is required.

The MICOM terminal switch has been extended to its maximum capacity: 17 lines to computers and 128 lines to terminals. It is not intended to make any further investments in this particular area, since this functionality can in future be provided at lower cost using Ethernet.

An investigation was made into the origins of electromagnetic interference, which perturbed in particular the instruments D1A and D11. The cabling was reorganised with better screening, producing a significant improvement.

# CENTRAL COMPUTING SERVICE

## OPERATION OF THE DEC-10 AND VAX 8650

(C. Rey)

The DEC-10 has been used at an almost constant level for interactive work, essentially by visitors. The batch work, which was still considerable at the beginning of the year, was gradually transferred to the VAX, particularly where larger jobs are concerned, in order to make the best use of the power of the new machine. As regards the instrument data, we have continued to transfer these to the DEC-10 for most of the instruments ; this has ensured continuous service for the treatment of this data with minimum disturbance.

The VAX 8650 started to receive data from the instruments with the 2nd reactor cycle this year. From that date the ILL scientists were able to transfer and adapt the applications directly associated with the treatment of the raw data. The load on the VAX was very high for the whole of the year with a mean utilisation of 400 to 500 hours over a period of 5 months. The interactive part continued to increase until August, to reach a maximum of 200 hours/month.

As regards the reliability of the actual hardware, it may be noted that for the DEC-10, there were 2 periods of 15 days and one month, when there were occasional problems at the level of the CPU ; despite this, the availability of the machine was of the order of 98%. There has been no particular problem on the VAX 8650, and its availability has been close to 100%.

## PROJECT FOR REPLACEMENT OF THE DEC-10

(M. Le Sourne)

The VAX 8650 is a machine shared by ILL and ESRF for use in the ratio 2:1. Since the end of the first half of 1987, ILL has used more than 66% of the power of this VAX, and could therefore not meet the slightest requirement for additional power in the future. In addition, we had strong arguments for closing down the DEC-10, viz:

- high maintenance cost
- lack of software on this type of machine; developments by DEC were stopped after announcement of the termination of the DEC-10/DEC-20 series
- difficulty of operating two different systems with a staff reduced in preceding years.

The Computing Department prepared a project for the replacement of the DEC-10 by a new machine to be used in joint operation with the VAX 8650. After consulting European manufacturers and some American firms, the ILL Management submitted to the Steering Committee documents proposing the purchase of a VAX 8700, to be installed in a cluster with the VAX 8650. This proposal was accepted at the Steering Committee meeting on 26 November.

## SYSTEM AND COMMUNICATIONS

(O. Tillier)

Activity in this field was largely devoted to improving all the products permitting the Central Computing Service to control the operation of the VAX:

- for the actual system: setting up new procedures for safeguarding and restoring files, monitoring and optimization of the VMS system in the light of the considerable increase in workload, testing prospective archiving packages, etc...

- for the exploitation of data: organization of the data received from instruments, provision of a number of software tools to receive these data, and to archive, verify and utilize them with the aid of application programs, to stock them on different types of supports for transfer to other computing centres.

- for the use of external networks: development of procedures to provide statistics of use, monitoring, security of access to our machines, keeping up to date the data base of the nodes accessible in the various networks such as JANET, EARN, BITNET and others.

Two Macintosh workstations, and a laser printer linked by Apple talk network to other Macintosh machines have also been installed for self service operation. This installation enables scientists to print copies of graphs from the screen and to produce documents for publication combining text and high quality graphs.

## GRAPHICS

(Y. Lefebvre)

Here too a major effort has been devoted to improving and documenting the items introduced last year, developing new graphic interfaces to permit the scientists to convert their DEC-10 applications to the VAX in a better context of compatibility and to take more advantage of the new graphic standards:

- modification of the Tektronix PLOT-10 library
- re-writing of DEC-10 subroutines to the GKS standard
- considerable assistance effort: documentation, provision of a "Tutorial program", assistance and information directly to scientists for writing their graphics applications.

The graphics group has also prepared a new version of the SCHAKAL program for the JUPITER colour terminal, and has developed an image preparation phase in order to exploit SCHAKAL more efficiently (SCHAKAL is a program which allows the generation of realistic drawings of molecules and crystallographic models).

A new software, CHEM-X, was installed on the VAX for use on the EVANS & SUTHERLAND workstation and the JUPITER terminal. This is a series of "molecular modelling programs".

A VAX-GPX graphics workstation has been introduced to enable us to treat more efficiently certain applications normally dealt with on the Jupiter terminal, and which demand a great deal of local intelligence.

## **MATHEMATICS**

### **(C. de Polignac)**

The NAG library with single and double accuracy has been installed on the VAX as a sharable code. An effort has been made to extend the on-line documentation.

Symbolic calculation applications have shown the limits of the "REDUCE" language, and have led us to study the product "MACSYMA".

A software for finite elements (MODULEF) has also been introduced.



**ADMINISTRATION DEPARTMENT**

## EINLEITUNG

Aus dem allgemeinen Verwaltungsbereich ist für 1987 folgendes berichtenswert:

- Die Planungen und Diskussionen zur Zusammenarbeit mit der ESRF nahmen einen breiten Raum ein. Die Direktoren beider Institute haben entschieden, dass ILL und ESRF auf einem gemeinsamen Forschungsgelände arbeiten sollen; ein dementsprechender Pachtvertrag wurde mit ESRF und CEA ausgearbeitet.

Mit der Räumung des künftig der ESRF vorbehaltenen Teils des bisherigen ILL-Geländes wurde begonnen. Die weiteren noch notwendigen Räumungsarbeiten sind geplant, werden sich aber wegen einiger noch zu lösender Probleme bis Mitte 1988 hinziehen.

- Der Abbau der 17,5 Stellen des Modernisierungsprogramms wurde Ende 1987 geschafft. Dies ist die Voraussetzung dafür, dass die Direktion von der Ermächtigung Gebrauch machen kann, die Zahl der Wissenschaftler um 4 zu erhöhen, soweit ebenso viele Stellen ausserhalb des wissenschaftlichen Sektors eingespart werden.

- Die Finanzbuchhaltung wurde von einem bisher externen EDV-gestützten System auf ein internes System umgestellt. Die Umstellungsarbeiten erwiesen sich als sehr arbeitsintensiv und langwierig; sie werden erst kommendes Jahr abgeschlossen werden können.

- Sorge bereitet 1987 die Uranversorgung. Die Export-Lizenzen der amerikanischen Behörden für 77,5 kg hochangereichertes Uran konnten wegen der neuerdings erforderlichen Einschaltung des amerikanischen Verteidigungsministeriums nur mit monatelanger Verspätung und Dank energischer Interventionen der EG-Versorgungsagentur erlangt werden. Anschliessend gab es weitere unvorhergesehene Verzögerungen wegen des vorgeschriebenen Sicherheits-Transports.

- Nachdem schon Spanien seit dem 1.1.1987 wissenschaftliches Mitglied am ILL geworden war, ist eine ähnliche Vereinbarung für die Zeit ab 1.1.1988 nun auch mit der Schweiz ausgearbeitet worden. Basis ist in beiden Fällen ein Zuschuss in Höhe von 1,5 % zu den Ausgaben des ILL.

- Der Lenkungsausschuss tagte im Juni 1987 in Grenoble und im November 1987 in Herstmonceux-Castle in Grossbritannien, dem Sitz des Royal Greenwich Observatory. Dort wurde das neue ILL-Budget für das Haushaltsjahr 1988 mit Gesamtausgaben von 284,6 Mio F beschlossen. Zugleich wurde die Erneuerung der zentralen Rechenanlage durch Beschaffung eines VAX 8700-Rechners einschliesslich Peripherie genehmigt.

## INTRODUCTION

Dans le domaine de l'Administration, parmi les points d'ordre général les points suivants méritent d'être mentionnés :

- les projets et les discussions concernant la collaboration avec l'ESRF ont tenu une grande place. Les Directeurs des deux Instituts ont décidé que l'ILL et l'ESRF devraient travailler sur un site scientifique commun ; un contrat de bail a été préparé en ce sens conjointement avec l'ESRF et le CEA.

On a commencé à dégager la partie du site de l'ILL dès lors réservée à l'ESRF. Des travaux pour libérer le reste du terrain ont été planifiés, mais leur réalisation s'étendra encore jusqu'à mi-1988 en raison de différents problèmes à résoudre.

- L'absorption des 17,5 cases prévues dans le cadre du Plan de Modernisation a abouti fin 1987. C'est la condition préalable pour la Direction de l'ILL de pouvoir augmenter de quatre le nombre de scientifiques, pourvu que le même nombre de postes soit supprimé en dehors du secteur scientifique.

- La comptabilité a été transférée d'un système assisté par ordinateur externe à un système interne. Cette conversion a nécessité beaucoup de temps en personnel et s'est avéré être un travail de longue haleine. Ce travail ne sera terminé que durant l'année à venir.

- En 1987 la fourniture d'uranium a soulevé quelque inquiétude. Il n'a été possible d'obtenir les licences d'exportation par les autorités américaines pour 77,5 Kg d'uranium hautement enrichi qu'après des mois de retard et grâce à l'intervention énergétique de l'Agence d'Approvisionnement de la Communauté Européenne, à cause de l'obligation récente nécessitant l'intervention du Ministère de la Défense des Etats-Unis. Ensuite il y a eu d'autres retards imprévus dus au transport en conditions de sécurité.

- A la suite de la participation scientifique de l'Espagne à l'ILL depuis le 1.1.1987, un accord semblable a été passé avec la Suisse avec effet au 1.1.1988. Dans les deux cas, cet accord est basé sur une contribution de 1,5 % aux dépenses de l'ILL.

- Le Comité de Direction s'est réuni en juin 1987 à Grenoble, et en novembre 1987 au Château de Herstmonceux en Grande-Bretagne, où se trouve l'Observatoire de Greenwich. C'est là que le nouveau budget ILL pour l'année 1988 a été adopté avec une enveloppe globale de 284,6 MF. Par la même occasion, le renouvellement de l'ordinateur central par l'achat d'un ordinateur VAX 8700 comprenant les périphériques, a été approuvé.

## INTRODUCTION

The following items may be selected for mention in the general Administration area for 1987:

- The planning and discussions on cooperation with the ESRF were very comprehensive. The Directors of the two institutes have decided that ILL and ESRF should operate on a common research site; a contract of lease in this sense was prepared jointly with ESRF and CEA.

A start was made on clearing that part of the ILL site reserved in future for the ESRF. Plans have been made for the remaining clearance work required, but this will only be completed in mid-1988, due to various problems to be resolved.

- The reduction of the staff complement by the 17.5 posts under the Modernisation Programme was completed by the end of 1987. This is a precondition for the ILL Management's making use of the authorisation to increase the number of scientists by four, provided that the same number of posts can be economised outside the scientific sector.

- Bookkeeping was transferred from an external computer-assisted system to an internal system. The conversion work proved extremely labour-intensive and slow. This work will only be completed in the coming year.

- In 1987 the uranium supply caused concern. It was only possible to obtain the export licences from the American authorities for 77.5 Kg highly enriched uranium after months of delay and thanks to the energetic assistance of the EC Supply Agency, because of the recent requirement to obtain the approval

of the US Ministry of Defence. Subsequently there were further unforeseen delays, due to the mandatory secure shipment.

- Following the Spanish scientific membership of ILL since 1.1.87, a similar agreement has now been reached with Switzerland with effect from 1.1.88. In both cases this is based on a contribution of 1.5 % to the ILL's expenditure.

- In June 1987 the Steering Committee met in Grenoble, and in November 1987 at Herstmonceux Castle in Sussex, where the Royal Greenwich Observatory is situated. There the new ILL budget for the year 1988 was adopted, with total expenditure of 284.6 MF. At the same time the renewal of the central computer by the purchase of a VAX 8700 computer including peripherals was approved.

## FINANCE

### Implementation of 1987 Budget (Normal Budget)

The budget authorized for 1987, including 9.4 MF brought forward from 1986, amounted to total expenditure of 286.2 MF excluding taxes, which was financed as follows:

	MF
Balance of expenditure 1986 .....	4.6
ILL's own income .....	10.5
Spanish contribution .....	4.1
Associates' contributions I .....	267.0
	<u>282.2</u>
Associates' contributions I .....	267.0
less amount brought forward .....	9.4
Associates' contributions II .....	<u>257.6</u>

In 1987 there were 5 reactor cycles, and installation of the Cold Source was completed by the end of November.

This resulted particularly in savings in the reactor area, so that when the 1987 inflation rate is taken into account there is only a relatively small increase in operation expenditure in comparison with 1986.

To cover the return to full reactor operation, the purchase of 25 kg uranium and the manufacture and delivery of 6 fuel elements were planned for 1987 as well as the reprocessing of 4 fuel elements. This resulted in a considerably higher planned expenditure for fuel elements in comparison with 1986, so that the total operation expenditure in the Budget increased to 238.0 MF (217.5 MF expenditure in 1986).

The total investments were increased by approximately 22.8 %, particularly to cover the costs resulting from the manufacture of instruments for the Cold Source.

### Modernisation Programme

Of the carry forward approved to 1986 of 15.9 MF, a balance of expenditure of 8.99 MF was again carried forward to the financial year 1987.

With the start of the installation of the Cold Source in November 1987, the great majority of these funds brought forward to 1987 have now been paid out to the suppliers.

The cumulative total expenditure including the sum brought forward from 1986 thus amounts to 147.9 MF.

## FORWARD LOOK

The ILL's investment plans in the scientific field are essentially concentrated on:

- completion of the instruments for the Horizontal Cold Source (IN14, IN10C, IN15 and D22) with expenditure amounting to 8.2 MF in 1988 and approximately 3.6 MF in 1989. The commissioning of IN10C, IN15 and D22 is planned for 1989/90.

## Administration Department

- the renewal, already decided, of the central computer by a more powerful machine (VAX 8700), to satisfy the increased computing requirements for the future. The financing of this continues into 1989.

- the establishment of facilities used jointly with ESRF, to find a practical, advantageous and economic procedure for cooperation in the interests of science.

- the replacement of obsolescent installations and instruments, to maintain the safety of the Reactor operation and the competitiveness of the Institut.

For these new investments a constant investment figure totalling approximately 31.5 MF is planned for the next few years, although these figures are based only on a minimum figure for renewal of obsolescent instruments.

### Comparison between Normal Budget 1986 and 1987

	Expenditure 1986		Revised 1987 Budget excl. carry forward from 1986		Carry forward from 1986	Revised 1987 Budget
	MF	%	MF	%	MF	MF
1) Staff costs	145.1	58.2	149.2	53.9	1.9	151.1
Other exp. on personnel	1.7	0.7	1.7	0.6	—	1.7
2) Fuel Elements	18.4	7.4	31.9	11.5	2.0	33.9
Consumables	24.9	10.0	26.7	9.7	1.0	27.7
Long term supplies and services	7.8	3.1	8.9	3.2	—	8.9
Short term supplies and services	9.9	4.0	9.5	3.4	—	9.5
Transport	1.8	0.7	1.8	0.7	—	1.8
Misc. Admin. costs	6.5	2.6	7.0	2.5	—	7.0
Taxes & fees	1.4	0.6	1.3	0.5	—	1.3
3) Operation	52.3	21.0	55.2	20.0		56.2
<b>I. Total operation</b>	<b>217.5</b>	<b>87.3</b>	<b>238.0</b>	<b>86.0</b>	<b>4.9</b>	<b>242.9</b>
Buildings	1.4	0.6	2.7	1.0		2.7
Equipment	3.7	1.5	5.2	1.9	3.3	8.5
Instruments	16.3	6.6	20.0	7.2	0.5	20.5
Other investments	10.2	4.0	10.9	3.9	0.7	11.6
<b>II. Investments</b>	<b>31.6</b>	<b>12.7</b>	<b>38.8</b>	<b>14.0</b>	<b>4.5</b>	<b>43.3</b>
<b>III. Total expenditure</b>	<b>249.1</b>	<b>100.0</b>	<b>276.8</b>	<b>100.0</b>	<b>9.4</b>	<b>286.2</b>

	Income 1986		Revised Budget 1987 excl. carry forward		Revised Budget 1987
	MF	%	MF	%	MF
Balance of expenditure	—	—	4.6	1.7	4.6
ILL's own income	8.9	3.5	10.5	3.8	10.5
Spanish contribution	—	—	4.1	1.5	4.1
Associates' contributions	240.2	96.5	257.6	93.0	267.0
<b>TOTAL</b>	<b>249.1</b>	<b>100.0</b>	<b>276.8</b>	<b>100.0</b>	<b>286.2</b>

## PURCHASING

The major components of the Horizontal Cold Source have been delivered and assembled; the source was tested in the reactor in December 1987. The helium compressor and refrigerator circuit have already been successfully tried out. The installation of the main neutron guides is on schedule and due for completion by CILAS ALCATEL in January 1988. The main shielding around H5 guides was built by REYDEL.

Some important parts have been ordered for the new instruments of the cold source. The shielding for the analyser and detector of IN14 will be constructed by MAINANCE Ltd. and IN14 should be ready for tests with cold neutrons in the third reactor cycle of 1988. The primary shielding for IN10C will be made by FONDERIE DE GENTILLY; SIGRI have been awarded the contract for the manufacture of the 22 metre long tube of the small angle spectrometer D22. More recently invitations to tender were sent out for the solenoid coils of IN15.

Other major purchases for the instruments include the neutron detectors for IN5 which were awarded to PECHINEY XERAM and the hybrid circuits for D20 which were ordered from SECRE.

Early in the year, the institute ordered 6 fuel elements from NUKEM and 6 from CERCA to be delivered during 1987-89. 32 kg of Uranium 235 will be purchased before the end of the year.

The gamma detection equipment which has been in operation since 1970 was replaced by new ambient air and water circuit meters from BERTHOLD. The Reactor interphone network will be replaced by STENTOFON equipment to be installed by BALAIN POULAT.

The Steering Committee approved the purchase from DIGITAL of a Vax 8700 computer which will replace the DEC 10 central computer. The Institute also invested in a Computer aided design (CAD) system supplied by MATRA DATAVISION which will be linked to the system at the ESRF.

The Institute ensured that firms in the member countries were invited to tender for all major purchases. This competitive tendering continues to bring considerable savings.

ILL is handling purchases for the ESRF; about 400 orders were placed including a CAD system from MATRA and a Rotating Anode X-ray Generator from ENRAF NONIUS. The purchasing procedures are in progress for the 200 MeV Electron Linac and the ESRF Industrial Architect.

The distribution of ILL's free purchases \* in 1987 is shown in the diagram.

Suppliers from	MF	%
France	10.5	38
UK	2.3	9
Germany	6.7	24
Others	8.0	29
<b>Total</b>	<b>27.5</b>	<b>100</b>

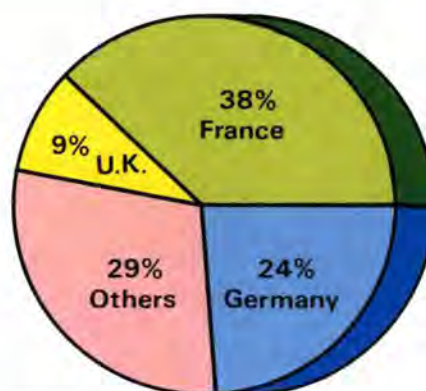


Fig. 108 Distribution of ILL's free purchases in 1987.

\* Free purchases means that a free choice of suppliers was possible excluding therefore the fuel cycle, electricity and small purchases of less than FF 50 000.

## STORES

All deliveries of purchased equipment were received and checked quantitatively by the storemen who at the same time executed the despatch of samples, returned equipment etc.

A number of new items were added to the range of articles held in stock and some of the less popular articles were removed reflecting the changing needs of scientists, technicians and other staff.

# PERSONNEL

## STAFF

The few figures which follow give a very partial image of the ILL staff. It may be noted that further details on social problems and personnel can be found in the 'Bilan Social' (personnel statistics), published every April and summarizing the situation for the previous three years.

In 1987 the authorized staff complement was reduced from 498 to 493 staff, including two Spanish thesis student posts.

The breakdown by nationalities is as follows:

French .....	65.14 %
German .....	14.74 %
British .....	12.65 %
Others .....	7.57 %

A reduction in German and British staff was noted in 1987. This was due partly to the fact that certain posts of German and British staff who left were not filled (plan for absorption of posts), and partly to the great difficulty in finding non-scientific staff in the two countries who meet our requirements and can be incorporated in the ILL salary scales, particularly as regards "non-cadres".

There was as usual a considerable fluctuation in the scientific personnel (21.13 %). This phenomenon should diminish in 1988.

The average age increased from 40 in 1986 to 40.79 in 1987 (41.79 for men and 39.80 for women).

As in 1986, 83 % of the staff were male.



W. Dowse (right) retired on 31 May 1987. He was presented with a gift from his colleagues by C. Eitner.

## Staff Movements (on posts) in 1987

CATEGORIES	POSITION on 31.12.86					DEPARTURES 87					RECRUTMENT 87					DIFFERENCE					SITUATION on 31.12.87					CHANGE (%) col. 2 compared to col. 1				
	D	B	F	Oth.	T	D	B	F	Oth.	T	D	B	F	Oth.	T	D	B	F	Oth.	T	D	B	F	Oth.	T	D	B	F	Oth.	T
<b>"CADRES"</b>																														
SCIENTISTS (Perm.)	12	9	7	3	31	—	3	—	—	3	—	1	—	—	1	—	—2	—	—	—2	12	7	7	3	29	—	33.3	—	—	9.68
ENGINEERS	—	—	2	—	2	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	—	2	—	2	—	—	—	—	—
SCIENTISTS (non perm.)	13	9	8	8	38	4	5	1	2	12	2	4	5	3	14	-2	-1	4	1	2	11	8	12	9	40	30.77	55.55	12.5	25	31.58
<b>TOTAL</b>	<b>25</b>	<b>18</b>	<b>17</b>	<b>11</b>	<b>71</b>	<b>4</b>	<b>8</b>	<b>1</b>	<b>2</b>	<b>15</b>	<b>2</b>	<b>5</b>	<b>5</b>	<b>3</b>	<b>15</b>	<b>-2</b>	<b>-3</b>	<b>4</b>	<b>1</b>	<b>—</b>	<b>23</b>	<b>15</b>	<b>21</b>	<b>12</b>	<b>71</b>	<b>16</b>	<b>44.44</b>	<b>5.88</b>	<b>18.18</b>	<b>21.13</b>
OTHER "cadres"	14	10	43	—	67	1	2	—	—	3	—	—	—	—	—	-1	-2	—	—	-3	13	8	43	—	64	7.14	20	—	—	4.48
<b>TOTAL "cadres"</b>	<b>39</b>	<b>28</b>	<b>60</b>	<b>11</b>	<b>138</b>	<b>5</b>	<b>10</b>	<b>1</b>	<b>2</b>	<b>18</b>	<b>2</b>	<b>5</b>	<b>5</b>	<b>3</b>	<b>15</b>	<b>-3</b>	<b>-5</b>	<b>4</b>	<b>1</b>	<b>-3</b>	<b>36</b>	<b>23</b>	<b>64</b>	<b>12</b>	<b>135</b>	<b>12.82</b>	<b>35.71</b>	<b>1.66</b>	<b>18.18</b>	<b>13.04</b>
THESIS STUDENTS	10	8	8	1	27	2	—	5	1	8	1	—	3	3	7	-1	—	-2	2	-1	9	8	6	3	26	20	—	62.5	100	29.63
<b>"NON-CADRES"</b>																														
TECHNICIANS	16	27	123	2	168	1	1	—	—	2	1	—	—	—	1	—	-1	—	—	-1	16	26	123	2	167	6.25	3.70	—	—	1.19
OTHERS	9	12	126	13	160	2	2	2	1	7	2	—	3	—	5	—	-2	1	-1	-2	9	10	127	12	158	22.22	16.6	1.59	7.6	4.37
<b>TOTAL "non cadres"</b>	<b>25</b>	<b>39</b>	<b>249</b>	<b>15</b>	<b>328</b>	<b>3</b>	<b>3</b>	<b>2</b>	<b>1</b>	<b>9</b>	<b>3</b>	<b>—</b>	<b>3</b>	<b>—</b>	<b>6</b>	<b>—</b>	<b>-3</b>	<b>1</b>	<b>-1</b>	<b>-3</b>	<b>25</b>	<b>36</b>	<b>250</b>	<b>14</b>	<b>325</b>	<b>12</b>	<b>7.69</b>	<b>0.80</b>	<b>6.66</b>	<b>2.74</b>
<b>GRAND TOTAL</b>	<b>74</b>	<b>75</b>	<b>317</b>	<b>27</b>	<b>493</b>	<b>10</b>	<b>13</b>	<b>8</b>	<b>4</b>	<b>35</b>	<b>6</b>	<b>5</b>	<b>11</b>	<b>6</b>	<b>28</b>	<b>-4</b>	<b>-8</b>	<b>3</b>	<b>2</b>	<b>-7</b>	<b>70</b>	<b>67</b>	<b>320</b>	<b>29</b>	<b>486</b>	<b>13.51</b>	<b>17.33</b>	<b>2.52</b>	<b>14.81</b>	<b>7.1</b>

(Figures in blue are negative).

## **SALARIES**

In 1987 there was an overall salary increase of 2.1%.

As at the CEA, advancements represent 1.5 % and seniority 0.35 % of the total payroll.

## **GUEST SCIENTISTS**

In 1987, 1184 guest scientists came to ILL, including 387 French, 243 British, 338 German and 216 of other nationalities.

This represented 1995 visits in 1987, slightly more than in 1986 (1950) despite the reduction in the number of cycles (5 instead of 6).

## **ILL/ESRF COLLABORATION**

The ILL personnel group acts on behalf of ESRF as regards offers of employment, payment of salaries, with the "PACHA" computer system, and travel expenses (Missill system). This covers 32 ESRF employees at present.

## **WELFARE AND GENERAL SERVICES**

### **HOUSING**

#### **1) Employers' contribution to housing**

The housing situation in Grenoble has become a little easier during the last few months. However rents are high - Grenoble is generally considered as being the third French town, after Paris and Nice, as regards housing costs.

As a result, we receive requests from staff:

for housing at reasonable cost (HLM type):

In 1987, 6 families were rehoused, either in flats or houses - at a relatively low cost in relation to market prices: e.g. 1 600 F for two rooms plus kitchen and bathroom. Average ILL subsidy: 49 000 F per flat or house.

for loans:

15 loans at an average of 53 000 F each (a total of 741 900 F was allocated in 1987, which brings to 259 the number of loans granted since the foundation of the ILL under the law requiring employers to make a contribution to employees' housing.

#### **2) New Families**

In 1987 we helped 25 persons (new ILL or ESRF employees and long-term visitors) to find accommodation. We estimate an average of 6 hours' work per person assisted (telephone calls, visits, accompanying them for signature of contracts, etc.).

#### **3) Guest Scientists**

We have 20 furnished flats, which were rented to 150 ILL or ESRF scientists on short visits.

## **INTERNATIONAL EDUCATION**

There have been considerable developments in recent months.

#### **1) "Houille Blanche" Primary School**

There are now almost 150 pupils, of whom approximately 50 % are French, and 50 % of German or English mother tongue.

Two other primary schools either include or will shortly have "international sections":

The "Jean Jaurès" school opened an Italian section in September 1987 ; The "Clémenceau" school has a German section.

### 2) Lycée International Stendhal

The former Stendhal Secondary school and Lycée, in the centre of Grenoble, has been converted into the Lycée International Stendhal (LIS).

Since September 1987 there have been international sections in this school for the classes "sixième" (age 11) and "seconde" (age 15). Additional international classes will be introduced each year until international classes are available throughout the school from September 1990.

What is the purpose of these "sections" ? To lead bilingual children with two cultural backgrounds up to the "baccalauréat" of their choice. Instruction is given by teachers from the country of origin in language, literature, history and geography in the mother tongue for seven and a half hours per week at age 11 - seven hours at age 15. This appears to fit in well with the general syllabus of the French Ministry of Education.

### 3) "Eaux Claires" CES and Lycée

The "sections internationales" at the Eaux Claires are being progressively closed as the classes are transferred to the LIS, which inevitably makes the remaining classes increasingly marginal; in particular this results in difficulties in finding teachers prepared to teach only a few hours per week of German or English to the non-French pupils.

### 4) Teachers' Salaries

In the past the ILL has financed the whole of the mother tongue education for German and British pupils in the schools concerned.

The German and Italian governments have agreed to accept responsibility for the payment of teachers of their nationality with effect from September 1987. Discussions are in progress with the British authorities with a view to reaching a similar solution.

The Directors of the ILL and ESRF are following with interest the present experiment, which certainly represents progress in comparison with previous years.

## Training

In 1987 there were 12 000 hours of training (on average 50 hours per employee concerned).

The subjects include:

- languages (French, English, German)
  - courses prepared for ILL staff, for example:
    - industrial design for experiment technicians ;
    - introduction to Algorithm and to the language Basic for engineers and technicians ;
    - a course for scientists entitled "Linear reflexions on poisson type data".
  - an advanced course on word-processing, particularly for the secretaries in the scientific area, is given by one of the secretaries at ILL who has specialised in this specific field.
  - technical courses associated with particular jobs.
- Each year the Training Office receives almost 200 requests for

training. The Training Programme is prepared in close collaboration with the Heads of Departments and Groups, and with the Training Subcommittee of the Works Committee.

## REPROGRAPHY

Last year we reported that the ILL Reprography Workshop was now capable of dealing with requests for a variety and an increasing number of jobs, previously done outside ILL, thanks to its equipment and to the skill of its two part time staff.

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**MISCELLANEOUS**

# LIST OF SEMINARS

## COLLEGE 2

### Theory

"Une solution diagrammatique du modèle d'Anderson périodique".  
R. IGLESIAS, Université de Porto Alegre (Brésil).

"Thee effective dielectric constant of a dispersion of spheres".  
U. GEIGENMÜLLER, Institut Lorentz, Leiden (Pays-Bas).

"Collective behaviour of field induced spin density waves in Bechgaard salts".  
K. MAKI, MPI, Stuttgart.

"Conductivity in the lowest Landau level of a disordered electron system".  
W. APEL, MPI, Stuttgart.

"Superlocalisation au voisinage du seuil de percolation: évidence expérimentale".  
G. DEUTCHER, Univ. de Tel-Aviv.

"Kondo effect: from Fermi liquid to scaling".  
P. WIEGMANN, Institut Landau, Moscow.

"Survey of some classical and quantum phenomena at crystal surfaces".  
V. MARCHENKO, Institut de Physique des Solides, Chernogolovka.

"Theory of drifting Néel lines in oscillating fields".  
V. MARCHENKO, Institut de Physique des Solides, Chernogolovka.

"Superconductivity and charge ordering in systems with local electron pairs".  
S. ROBASZKIEWICZ, ILL.

"Small polaron and bipolaron formation".  
A. LAGENDIJK, Université d'Amsterdam.

"Hydrodynamic boundary conditions and cooling in superfluid  $^3\text{He}$ ".  
M. LIU, University of Hannover.

"Incommensurate charge density waves: internal symmetries and solitons".  
B.K. CHAKRAVERTY, LEPES, CNRS-Grenoble.

"Un nouvel état de la matière : les conducteurs de basse dimensionnalité sous champ magnétique. Le cristal ultraquantique".  
P. LEDERER, Laboratoire de Physique des Solides, Orsay.

"Vortices in the dynamic form factor of two dimensional easy plane magnets".  
F.G. MARTENS, University of Bayreuth.

"Inhomogeneous phases in colloidal fluids".  
T. SLUCKIN, ILL.

"Effective Fermi liquid theory for disordered electrons".  
C. CASTELLANI, Università di Roma.

"Possibilité d'observation par RMN de la violation de la parité en physique moléculaire".  
A.L. BARRA, SNCI, CNRS-Grenoble.

"Quelques problèmes physiques d'électro-chimie".  
T.C. HALSEY, SPHT, Saclay.

"Correlated diffusion in solids: problems concerning random networks".  
R.A. TAHIR-KHELI, Temple University, Philadelphia.

"L'anapole : un curieux moment magnétique impair et ses effets en physique atomique et moléculaire".  
C. GIGNOUX, I.S.N.-Grenoble.

"Ground state properties of quantum spin chains".  
J. SOLYOM, ILL.

"Channelling and quantum chaos".  
R. PRANGE, University of Maryland.

"Critical threshold dynamics of a sliding CDW".  
L. SNEDDON, Brandeis University and ILL.

"Non-linear fluctuation effects in 1-d quantum spin chains".  
D. HALDANE, AT&T Bell Laboratories, Murray-Hill.

"Une nouvelle route vers le chaos".  
C. TSALLIS, CRTBT, CNRS-Grenoble.

"Exponents of surface roughness for stochastic growth (Eden Models)".  
D. WOLF, Université de Cologne.

"Quasicrystals: symmetry and stability".  
D. MUKAMEL, Dept. of Physics, Weizmann Institute, Rehovot.

"Quasiparticle interactions and anisotropic superconductivity in heavy fermions".  
Ch. VARMA, Bell Tel., Murray Hill, N.J.

"Spin induced superconductivity in  $\text{YBa}_2\text{Cu}_3\text{O}_{7-\delta}$ ".  
K. BEDELL, Los Alamos National Laboratory.

"Statistical mechanics of integrable models in 1 + 1 dimension".  
J. TIMONEN, Jyväskylä, Finland and Manchester, U.K.

"Collective modes in heavy fermion superconductors".  
J. RODRIGUEZ, ILL.

"On the stochastic transport in disordered systems".  
V. VINOKUR, Institut de Physique des Solides, Chernogolovka.

"Antiferromagnétisme et supraconductivité dans le modèle Hubbard bidimensionnel".  
H.J. SCHULZ, Orsay.

"Cubic phases in lyotropic liquid crystals".  
A. MAGGS, I.L.L.

"Transport properties of highly anisotropic superconductors".  
K. SCHARNBERG, University of Hamburg.

"Electron phonon coupling in intermetallic rare earth compounds".  
P. THALMEIER, ILL.

"Comment caractériser un arbre ? (un bassin de rivière, un agrégat colloïdal, un écoulement dans une argile, etc...)".  
J. VANNIMENUS, ILL et CNRS-Grenoble.

"Paires locales  $\bar{O}-\bar{O}$  et supraconductivité à  $T_c$  élevée".  
M. AVIGNON, CNRS-Grenoble.

"Strong coupling theory of high  $T_c$  superconductors".  
D. RAINER, University of Bayreuth.

## COLLEGE 3

### Fundamental and Nuclear Physics

"The neutron proton interaction and nuclear collectivity".  
R.F. CASTEN, Brookhaven National Laboratory.

"Interaction and annihilation of antiprotons and nuclei".  
T. VON EGIDY, TU München.

"Neutron activation analysis - an approach for solving some geological problems".  
G. VIVIER, Institut Dolomieu, Université de Grenoble.

"Measurement of the neutron half-life possible experimental improvements".  
L. BONDARENKI, Jurchatov Institute, Moscow.

"Capabilities of Central Bureau for Nuclear Measurements in nuclear target preparation".  
J. PAUWELS, CBNM, Geel.

"Status report on the neutron-antineutron experiment at ILL".  
T. BITTER, Phys. Inst. Heidelberg.

"BILL: Past - Present - Futur".  
B. KRUSCHE, K. SCHRECKENBACH, ILL.

"Ultrasensitive laser spectroscopy in nuclear structure physics".  
D.A. EASTHAM, Daresbury Laboratory.

“Evidence for pear-shaped nuclei”.  
P.A. BUTLER, University of Liverpool.

“Wanderings in Tibet”.  
J.A. PEARCE, University of Newcastle.

“Explosive helium burning in stars”.  
M. WIESCHER, Notre Dame, Indiana.

“Neutron radiative capture mechanism”.  
YU KUN HUO, Fudan University, Shanghai.

“Measurements on the neutron beta-decay with PERKEO”. J. LAST, University of Heidelberg.

“The ignored discovery of the element  $Z = 43$  and its implication for the discovery of fission”.  
P. VAN ASSCHE, Mol, Belgium.

“Neutral pion photoproduction at the Mainz microtron”.  
H. STRÖHER, University of Giessen.

“Search for a short-lived axion in neutron-proton capture”.  
J. DÖHNER, University of Heidelberg.

“Spin distributions in fission fragments”.  
J.L. DURELL, University of Manchester.

“Nanosecond lifetimes: quasiparticle excitations and collectivity in nuclei”.  
W. ANDREJTSCHJEFF, Sofia.

“The IBM for atoms, molecules and nuclei”.  
B.R. BARRETT, NSF Washington, D.C.

“g-factor measurements of Backbenders”.  
K.P. LIEB, University of Göttingen.

“Experimental studies of neutron emission for  $^{252}\text{Cf}(s,f)$  and fragmentation for  $^{235}\text{U}(n,f)$  in the resonance region”.  
C. BUDTZ- JORGENSEN (CBNM, Geel).

“Gamow-Teller transitions in nuclei close to  $^{100}\text{Sn}$ ”.  
I.S. GRANT, CERN.

“New developments in nuclear fission”.  
R. HASSE, GSI, Darmstadt.

“Neutron induced fission of  $^{235}\text{mU}$ ”.  
W.L. TALBERT Jr., Los Alamos.

“Time reversal tests with polarised neutrons”.  
P.K. KABIR, CERN.

“Dynamic deformation model and unified theory of nuclear structure, fission, alpha decay and superheavy nuclei”.  
K. KUMAR, Tennessee Tech.

“Facilities and programs in neutron activation analysis and gamma spectroscopy at ORNL”.  
F. DYER, Oak Ridge National Laboratory.

“Radiative muon capture on hydrogen”.  
G. AZUELOS, Triumf, Vancouver and University of Grenoble.

“A presentation of the instrument PN1 (Lohengrin)”.  
H. FAUST, ILL.

“New experiments with free polarized neutrons”.  
D. DUBBERS, ILL.

“A survey of eight years of nuclear supersymmetry”.  
J. JOLIE, ILL.

“Interferometry with pulsed neutrons”.  
H. WÖLWITSCH, Atominstutut Wien.

“Properties of the t-matrix and their effects in inelastic proton scattering”.  
K. AMOS, Melbourne University.

“Experiments with ultracold neutrons in the Leningrad Nuclear Physics Institute”.  
A. SEREBROV, Leningrad University.

“New developments in Ge detector technology”.  
A. SCHRÖDER, University of Köln.

## COLLEGE 4

### Structural and Magnetic Excitations

“Random fields, plastic crystals and melting”.  
S. GALAM, DRP, Université Paris VI.

“Measurement of charge-density wave velocity”.  
A. JANOSSY, Laboratoire de Spectrométrie Physique, Grenoble and KFKI, Budapest.

“Spin fluctuations and weakly magnetic materials”.  
S. HAYDEN, ILL.

“Superconductive and structural properties of alkaline-earth substituted  $\text{La}_2\text{CuO}_4$ ”.  
J.G. BEDNORZ, IBM-Zürich

“Dynamical studies on spin glasses by macroscopic techniques”.  
M. ALBA, ILL.

“Pinning by defects in incommensurate structures: irradiation experiments”.  
H. MUTKA, ILL.

“Neutron spin echo studies of spin waves in iron just below  $T_c$ ”.  
B. FARAGO, ILL.

“Localisation of vibrational modes in amorphous materials”.  
O. ENTIN-WOHLMAN, Tel Aviv University.

“Phase transition in ammonium sulfate and ferroelectricity”.  
A.ONODERA, Hokaido University, Sapporo (Japan).

“Reentrant spin glass behaviour in mixed garnets ( $\text{Fe}_x\text{Cr}_{1-x}$ ):  $\text{Ca}_3(\text{GeO}_4)_3$ ”.  
T. BRÜCKEL, University of Tübingen and ILL.

“Coexistence of f-instabilities and magnetic order”.  
A. SEVERING, ILL.

“Inelastic neutron scattering from the one-dimensional ionic conductor K-hollandite”.  
F. FREY, University of München.

“Neutron scattering in orientational glasses”.  
A. LOIDL, University of Mainz.

“Measurement of a phonon dispersion curve by inelastic X-ray scattering”.  
B. DORNER, ILL.

“Infrared and submillimetric spectroscopy of charge-density waves and TCNQ-salts”.  
V. ZELEZNY, Institute of Physics, Prague.

“Spallation neutron sources in Japan: today and future”.  
Y. ENDOH, Tohoku University, Sendai.

“Antiferromagnetic correlations and order in  $\text{UPT}_3$  and  $\text{U}(\text{Pt}_{.95}\text{Pd}_{.05})_3$ ”.  
P. FRINGS, ILL.

“Experimental observation of a two dimensional nuclear Heisenberg ferromagnet”.  
H. GODFRIN, ILL.

“Monochromator with variable horizontal and vertical curvature and its application”.  
W. BÜHRER, ETH-Würenlingen.

“Domain-wall phases of  $\text{D}_2$  on ZYX-graphite”.  
H.P. SCHILDBERG, ILL.

“Excitations in the commensurate and soliton phases of  $\text{D}_2$  on graphite”.  
V. FRANK, ILL and University of Mainz.

“Nuclear magnetic ordering of Cu”.  
M. STEINER, HMI, Berlin.

## COLLEGE 5

### Crystal and Magnetic Structures

“Complexes de coordination des radicaux. Libres nitroxydes : des Ferroaimants moléculaires ?”  
P. REY, LC, CEN-Grenoble.

“Topotaxy - A crystallographic phenomenon with chemical implications”.  
J.R. GÜNTER, University of Zürich.

“Condensed matter simulations on a highly parallel computer”.  
G.S. PAWLEY, University of Edinburgh.

“D20: available to external users”.  
P. CONVERT, ILL.

"3-dimensional structure of toxin II from the scorpion *Androctonus australis* Hector refined at 1.8 Å resolution".  
J. FONTECILLA, CRMC2, CNRS, Marseille.

"Spin density in 3d transition elements and their alloys".  
F. SACCHETTI, CNR-Frascati and University of Perugia.

"Démonstration de logiciel graphique moléculaire de (Chemical Design)".  
N. PERRY, S. BRIGGS, Chemical Design Ltd., Oxford.

"Neutron optical searches for violations of quantum mechanics".  
A.G. KLEIN, University of Melbourne.

"Crystal dynamics of complex crystals"  
K.R. RAO, NPD Barc Bombay.

"The study of collective excitations in crystals by pulsed neutron diffraction".  
B.T.M. WILLIS, Chemical Crystallography Laboratory, Oxford.

"Hydrogen bonding and molecular electrostatic potentials from diffraction data".  
B.M. CRAVEN, University of Pittsburgh.

"Direct triplet-phase measurements by means of multiple X-ray scattering".  
K. HÜMMER, University of Erlangen-Nürnberg.

"Triple-crystal diffraction studies on ion-implanted silicon crystals and other materials".  
S. WILKINS, CSIRO, Clayton, Australia.

"The effect of anion and cation doping on the magnetic ordering in  $RbFeCl_3$ ".  
D. VISSER, Oxford University.

" $^{35}Cl$  nuclear magnetic resonance, orthorhombic ammonium perchlorate ( $Pnma$ ?) and the possibility of phase transitions at subambient temperatures".  
S.SEGEL, Queen University, Kingston, Ontario.

## COLLEGE 6

### Liquids, disorder and defects in materials

"Photons and neutrons - Tools to investigate point defects in solids".  
W. TROST, ILL and MPI Stuttgart.

"Properties of collective modes in simple fluids and glasses".  
P.A. EGELSTAFF, University of Guelph (Canada) and Laboratoire Léon Brillouin, Saclay.

"Atomic transport and defects in metals".  
J. MUNDY, Argonne Nat. Lab. and Max Planck Institut für Metallforschung, Stuttgart.

"Dynamics of water as studied by neutron scattering techniques".  
J. TEIXEIRA, Laboratoire Léon Brillouin, Saclay.

"Hydrogen in Pd nanocrystals and alloys".  
Dr. SALOMONS, Free University of Amsterdam.

"Neutron scattering and molecular dynamics study of the pseudo-spin-glass RADP".  
H. GRIMM, Institut für Festkörperforschung, KFA Jülich.

"Statics and dynamics of quasiperiodic systems".  
T. JANSSEN, University of Nijmegen, (The Netherlands).

"Lattice dynamics of high  $T_c$  copper oxide superconductors".  
T. BRUN, Argonne National Laboratory.

"Dynamical properties of icosahedral, glassy and crystallized  $Pd_{58.8}Si_{20.6}U_{20.6}$ ".  
J.-B. SUCK, Kernforschungszentrum Karlsruhe, INFP.

"Electrical properties of small molecules".  
J. WILLIAMS, ILL.

"Liquid transition metal chalcogenides: the role of the transition metal d-electrons in determining the electrical and structural properties of these systems".  
A. BARNES, ILL.

## COLLEGE 8 and EMBL

### Biochemistry

"The interaction of RecA Protein with DNA".  
E. Di Capua (ETH-Zürich/ILL/EMBL).

"Guanin nucleotides and microtubule assembly".  
P. BAYLEY, National Institute for Medical Research, London.

"A molecular replacement solution for the structure of pro-insulin".  
Anne CLEASBY, Birkbeck College, London.

"New approaches in analysis and sorting of chromosomes".  
C. CREMER, Institut für angewandte Physik I der Universität, Heidelberg.

"The role of influenza virus hemagglutinin in membrane fusion".  
R.W.H. RUIGROK, National Institute of Medical Research, London.

"Structure and function of Rubisco".  
C. BRANDEN, Biomedical Center, Uppsala/EMBL/Heidelberg.

"Reflections on the higher order structure chromatin".  
H. EISENBERG, Weizmann Institut, Rehovot.

"The channel structure of Porin as determined by neutron diffraction".  
R.M. GARAVITO, University of Chicago.

"Le répresseur du système SOS chez *E. Coli*, LexA".  
M. SCHNARR, IBMC, Strasbourg.

"Viroids: plant subviral pathogens".  
T. CANDRESSE, INRA, Centre de Recherches de Bordeaux.

"Image reconstruction on RecA filaments".  
E. EGELMAN, University of Yale.

"Quaternary structure of the small subunit of the *E. Coli* ribosome".  
M. CAPEL, Brookhaven National Laboratory.

"Novel secretion of *E. Coli* hemolysin and DNA-binding proteins of *trypanosoma brucei*".  
C. HUGHES, Cambridge University.

"The use of quantum mechanical and molecular mechanical theoretical methods to study reaction processes in condensed phases (including proteins)".  
M. FIELD, Department of Chemistry, Harvard University.

"Genetic variation of *halobacterium halobium*".  
F. PFEIFER, Max-Planck-Institut für Biochemie, Martinsried.

"Supercoiled DNA- large scale opening and long-range effects".  
D. LILLEY, University of Dundee.

"The structure of alternating AT regions in supercoiled DNA".  
D. LILLEY, University of Dundee.

"Carbohydrate binding proteins (lectins) on tumor cells and their role in metastasation and drug targeting".  
F. CRAMER, Max-Planck-Institut für experimentelle Medizin, Göttingen.

"The crystal structure of a lipoprotein: The Lipovitellin: phospholipid complex".  
L. BANASZAK, St. Louis, Missouri.

"Genetic, biochemical and structural approaches of structure-function relationships of aminoacyl-tRNA synthetases".  
S. BLANQUET, Ecole Polytechnique, Paris.

"Strategies for the expression of plant viruses".  
A.-L. HAENNI, Institut Jacques Monod, Paris.

"Observation of acoustic Umklapp-Phonons in water-stabilized DNA by neutron scattering".  
H. GRIMM, Jülich.

"Three-dimensional study of viral pore-like structure".  
J.L. CARRASCOSA, Centro de Biología Molecular, Madrid.

"The crystal structure of methionyl-tRNA synthetase from *E. Coli*".  
Simone BRUNIE, Ecole Polytechnique, Palaiseau.

"Régulation artificielle de l'expression génomique par des oligonucléotides modifiés".  
C. HELENE, Museum d'Histoire Naturelle, Paris.

"Génétique moléculaire de la mitochondrie".  
P. SLONIMSKI, Centre Génétique Moléculaire, Gif sur Yvette.

"La leucyl tRNA synthetase de levure est impliquée dans l'excision des introns mitochondriaux".  
C. HERBERT et M. LABOUESSE, Centre Génétique Moléculaire, Gif sur Yvette.

"Génétique moléculaire de la mitochondrie".  
P. SLONIMSKI, Centre Génétique Moléculaire, Gif sur Yvette.

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C. HERBERT et M. LABOUESSE, Centre Génétique Moléculaire, Gif sur Yvette.

## COLLEGE 9

### Chemistry

"Dynamics at phase boundaries of blockcopolymers - A D-NMR-study".  
W. GRONSKI, University Freiburg.

"X-ray absorption spectroscopy, a tool for studying electronic structure and ordering of organic compounds: examples of Pyrrole, Thiophene and their polymers adsorbed on Pt".

G. TOURILLON, Université Paris-Sud, Orsay.

"Small angle neutron scattering studies of hydrating cement - observing the birth of a fractal system".

A. ALLEN, Harwell Laboratory.

"Molecular characteristics and solution properties of lightly charged associating polymers".

D.G. PEIFFER, Exxon research and Engineering Company, Annandale, New Jersey.

"Dynamics of molecular hydrogen complexes".

J. ECKERT, ILL and LANSCE, Los Alamos.

"Which information does one get from the osmotic deswelling of polymer gels?"

J. BASTIDE, Institut Charles Sadron, Strasbourg and Laboratoire Léon Brillouin, Saclay.

"Surface properties and electronic structure of amorphous  $Zr_{74}Ni_{26}$  and  $Zr_{74}Ni_{26}$  hydride studied by photo emission".

YU XIN-NAN, Chinese Academy of Science Beijing and ETH, Zürich.

"Applications and limitations of deuterium labeling methods to neutron scattering studies of polymers".

G. WIGNALL, Oak Ridge National Laboratory.

"Nonexponential correlation and relaxation functions in glassy systems - NMR studies".

E. RÖSSLER, Freie Universität Berlin.

"The processes of formation of order in liquid crystals".

Y. ISHIBASHI, Nagoya University Japan.

"Neutron, NMR, calorimetry and Raman studies of metal Hexamine compounds".

J.A. JANIK, Institute of Nuclear Physics, Krakow.

## THURSDAY COLLOQUIA

"Interfaces entre phases dans les composés d'insertion ionique. Rôle de la métrique fractale de l'interphase".

A. LE MEHAUTE, Laboratoire de Marcoussis.

"Elementary excitations in liquid  $^3He$ ".

R. SCHERM, Physikalische-Technische Bundesanstalt, Braunschweig.

"Electron spectroscopy - A many body problem".

K. SCHÖNHAMMER (University of Göttingen).

"Harmonic and anharmonic states in glasses".

W.A. PHILLIPS, Cavendish Laboratory.

"Static and dynamical aspects of the random field problem".

V. JACCARINO, University of California, Santa Barbara.

"Helium atom scattering studies of surface structure and vibrations".

J.P. TOENNIES, Max-Planck Institut für Strömungsforschung, Göttingen.

"Exotic nuclei"

Monique BERNAS, Institut de Physique Nucléaire, Orsay.

"Theory of the local tunneling spectrum of a vibrating adsorbate".

A. BARATOFF, IBM, Zürich.

"Neutron spin echo study of dynamic correlations near liquid-glass transition".

F. MEZEI, Hahn-Meitner Institut, Berlin.

"Low energy charge exchange collisions: a new spectroscopy".

S.L. BLIMAN, CEN-Grenoble.

"Studies of the structure, phase transitions, and dynamics of commensurate herringbone monolayers physisorbed on graphite".

H. TAUB, University of Missouri-Columbia.

"How to change the mass of a neutron".

A. ZEILINGER, Atominstytut der Österreichischen Universitäten, Wien.

"Structure of streaming fluids".

S. HESS, T.U. Berlin.

# I.L.L. WORKSHOPS AND CONFERENCES

## Organised by the I.L.L. in 1987

I.L.L., 23-25 September, 1987. Workshop on "Polymer Motion in Dense Systems". (organized jointly by the ILL and IFF-KFA/Jülich: U. Buchenau, B. Maier, A. Rennie, D. Richter, T. Springer). [Abstracts: ILL Technical Report 87IL08T].

## Published in 1987

I.L.L. 24-26 September, 1986. "Quantum Aspects of Molecular Motions in Solids", edited by A. Heidemann, A. Magerl, M. Prager, D. Richter, T. Springer. Springer Proceedings in Physics, Vol. 17 (Springer-Verlag, 1987)

## Sponsored by the I.L.L. and published in 1987

Grenoble, 7-11 July, 1986. ICAREA. International Conference on "Anomalous Rare Earths and Actinides. Valence Fluctuation and Heavy Fermions", edited by J.X. Boucherle, J. Flouquet, C. Lacroix, J. Rossat-Mignod (1987, North-Holland).

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## THESES

BALLOU R.:

Anisotropies magnétiques du cobalt dans les composés intermétalliques lanthanide-cobalt.

THESE, DOCTORAT D'ETAT, UNIVERSITE SCIENTIFIQUE ET MEDICALE DE GRENOBLE, OCTOBRE 1987.

CAVALEIRO DE MIRANDA P.M.:

Contributions to the search for a neutron EDM.

THESIS. UNIVERSITY OF SUSSEX, U.K., FEBRUARY 1987

GAY J.M\*:

Du bidimensionnel vers le tridimensionnel. Etude expérimentale par diffraction électronique et neutronique de systèmes moléculaires simples adsorbés sur graphite:

méthane, tétrafluorométhane et éthane.

THESE DE DOCTORAT D'ETAT, UNIVERSITE D'AIX-MARSEILLE II, SEPTEMBRE 1987

GUENSTER S.:

Neutronenbeugung am flüssigen System  $\text{CCl}_4/\text{CS}_2$ .

DIPLOMARBEIT, MATHEMATISCH-NATURWISSENSCHAFTLICHEN FAKULTÄT DER RHEINISCH-WESTFÄLISCHEN TECHN.HOCHSCHULE, AACHEN, NOVEMBER 1987.

IBEL K.:

Biophysikalische Anwendungen der Neutronen-Kleinwinkelbeugung. (Version Janvier 1987).

HABILITATIONSSCHRIFT, UNIVERSITÄT OSNABRÜCK, 1987

JOHNSON S.M.:

Paramagnetic scattering studies of metallic magnetism using polarised neutrons and polarisation analysis.

THESIS, UNIVERSITY OF SHEFFIELD, SEPTEMBER 1986

LOUCA P.:

From x-ray structure factors to electron-density distributions.

THESIS, UNIVERSITY OF BRISTOL, SEPTEMBER 1986.

MORE B.R.:

Directorial correlation studies of  $^{156}\text{Gd}$ ,  $^{158}\text{Gd}$ ,  $^{168}\text{Er}$  and  $^{126}\text{Te}$ .

THESIS, UNIVERSITY OF SUSSEX, NOVEMBER 1986.

PATTERSON C.J.F. :

Topographic, ultrasonic and diffraction studies of helical antiferromagnets.

THESIS, UNIVERSITY OF HULL, DECEMBER 1986.

TOUDIC B\*:

Etude par diffusion incohérente de neutrons et par résonance magnétique nucléaire de la dynamique individuelle et des transitions structurales dans les p-polyphényles.

THESE DE DOCTORAT D'ETAT, UNIVERSITE DE RENNES I, MARS 1986.

WEIKARD H.:

Messung von Beta-Zerfallsenergien kurzlebiger, neutronenreicher Atomkerne im Massenbereich  $101 < A < 106$  und  $A = 109$ .

THESIS, TECHNISCHE UNIVERSITÄT CAROLO-WILHELMINA ZU BRAUNSCHWEIG, 1986.

\* Experiments performed at the I.L.L.

# LIST OF PUBLICATIONS 1987

## INTERNAL REPORTS

T = Technical Reports  
G = General Reports

### 87MA1G

MAIER B., [Ed.]:  
Institut Max von Laue-Paul Langevin. 20. Issued on the occasion of the 20th anniversary of the Institut's foundation.  
GENERAL REPORT

### 87TA2G

TASSET F. :  
Manipulation d'échantillons transuraniens contenus dans des enceintes doubles parois collées.  
GENERAL REPORT. (RAPPORT DE SYNTHESE DU GROUPE DE TRAVAIL EMANANT DE LA CIS DU 16.10.1985)

### 87CA3G

CASTETS C. [Ed.]:  
1986 ILL Experimental Reports and Theory College Activities.  
GENERAL REPORT

### 87SA4G

SARKISSIAN B.V.B. :  
Progress report on IN11B. The multidetector neutron spin echo spectrometer.  
GENERAL REPORT

### 87K15T

KIM H.J.:  
WLF. Modified data fitting program of WLI.  
TECHNICAL REPORT

### 87RO6T

ROBERT A. :  
Focalisation des neutrons lents par un hexapole magnétique ; calcul des écarts à la norme idéale introduits par un instrument cryogénique réel.  
TECHNICAL REPORT.

### 87BR7T

BROCHIER D. :  
Etude statique et dynamique des écrans thermiques dans les cryostats et cryo-fours.  
TECHNICAL REPORT

### 87IL8T

ILL, IFF-KFA:  
Workshop on polymer motion in dense systems, ILL, Grenoble September 23-25, 1987. Abstracts.  
TECHNICAL REPORT

### 87HE9T

HEWAT A.W. :  
ILL-ISIS High resolution powder diffractometer comparison.  
TECHNICAL REPORT

### 87KO10T

KOLLMAR A., LARTIGUE C., DOUCHIN F. :  
Proposition for the choppers for the TOF-mode of IN15/ILL.  
TECHNICAL REPORT

### 87HE11T

HEIDEMANN A., ECKERT J. :  
Proposal for a high resolution spectrometer for cold neutrons at LANSCE.  
TECHNICAL REPORT.

### 87WU12T

WUTTKE J. :  
Program DEORIE - Fit of quasielastic spectra by different scattering laws, given as the intermediate scattering law  $G(q,t)$ , or given in energy space as  $S(q,\omega)$ .  
TECHNICAL REPORT

### 87GO13T

GOEBEL H. :  
Polarisation de spin et son analyse sur le spectromètre à rétrodiffusion IN13. Spin polarisation and analysis on the backscattering spectrometer IN13.  
TECHNICAL REPORT.

### 87RO14T

RODRIGUEZ J., ANNE M., PANNETIER J. :  
STRAP. A system for time-resolved data analysis (powder diffraction patterns).  
TECHNICAL REPORT.

### 87PR15T

PRAGER M., HEIDEMANN A. :  
Tunneling spectroscopy. Compilation of available data.  
TECHNICAL REPORT

### 87GO16T

GOEBEL H. :  
L'utilisation de neutrons polarisés sur un spectromètre à rétrodiffusion.  
TECHNICAL REPORT. RAPPORT DE STAGE, JUILLET-SEPTEMBRE 1986, ILL.

### 87BU17T

BUSSIERE A., CHAUVIN J., GRIVOT P., KOSSAKOWSKI R., LIAUD P., SAINTIGNON P., DE, SCHRECKENBACH K. :  
Study of some properties of gas mixtures with small neutron capture cross section.  
TECHNICAL REPORT

### 87LI18T

LINDNER P., HESS S. :  
ANISK, a program for anisotropic treatment of D11/D17 - SANS multidetector data.  
TECHNICAL REPORT.

### 87FI19T

FILHOL A. :  
RAFIN, RAFNB, RAFDB, RAFD19. Programs for the refinement of the: [UB] matrix, wavelength, cell parameters, instrument zero shifts in a single crystal diffraction experiment.  
TECHNICAL REPORT

### 87AN20T

ANTONIADIS A., BERRUYER J., FILHOL A. :  
Affinement de diagrammes de diffraction de poudre par la méthode du maximum de vraisemblance. Deuxième édition. (Version révisée du rapport 86ANO2T).  
TECHNICAL REPORT

### 87SO21G

SOUBEYROUX J.L. :  
D1B user guide. The Banana detector.  
GENERAL REPORT

### 87AN22T

ANTONIADIS A., BERRUYER J., FILHOL A. :  
ABFfit: Manuel de la version 3.0 pour le MacIntosh.  
TECHNICAL REPORT

## PAPERS PUBLISHED IN SCIENTIFIC PERIODICALS, BOOKS AND CONFERENCE PROCEEDINGS, 1987

### 87CI101

CIZEWSKI J.A., COLVIN G.G., BOERNER H.G., HOYLER F., KERR S.A., SCHRECKENBACH K. :  
Identification of higher representations of the  $U(6/4)$  supersymmetry in the nucleus  $^{193}\text{Ir}$ .  
PHYSICAL REVIEW LETTERS VOL. 58, PP. 10-12 (1987)

### 87CH102

CHOI J., JOSE J.V. :  
Correlation effects in  $S = 1/2$  random-exchange Heisenberg antiferromagnetic chains.  
PHYSICAL REVIEW B VOL. 35, PP. 419-422 (1987).

### 87WH103

WHITE D.H., BOERNER H.G., HOFF R.W., SCHRECKENBACH K., DAVIDSON W.F., EGIDY T.VON, WARNER D.D., JEUCH P., BARREAU G., KANE W.R., STELTS M.L., CHRIEN R.E., CASTEN R.F., LANIER R.G., LOUGHEED R.W., KOUZES R.T., NAUMANN R.A., DEWBERRY R. :  
Nuclear structure of  $^{231}\text{Th}$  from neutron capture and (d,p) reaction measurements. PHYSICAL REVIEW C VOL. 35, PP. 81-110 (1987)

- J87BU104**  
 BUSSIERE A., FELTIN D., HAMELIN B., JACOB E., KURODA K., LIAUD P., MICHALOWICZ A., NEMOZ C., SILLOU D.:  
 A photoelectronic image device for slow neutrons.  
 NUCLEAR INSTRUMENTS AND METHODS IN PHYSICS RESEARCH A VOL. 254, PP. 215-218 (1987)
- 87BE105**  
 BETHKE J., DOLINO G., ECKOLD G., BERGE B., VALLADE M., ZEYEN C.M.E., HAHN T., ARNOLD H., MOUSSA F.:  
 Phonon dispersion and mode coupling in high-quartz near the incommensurate phase transition.  
 EUROPHYSICS LETTERS VOL. 3, PP. 207-212 (1987)
- 87BO106**  
 BOEUF A., CACIUFFO R., PAGES M., REBIZANT J., RUSTICHELLI F., TABUTEAU A.:  
 Neutron diffraction study of the  $U_{1-x}Np_xO_2$  fluorites.  
 EUROPHYSICS LETTERS VOL. 3, PP. 221-227 (1987)
- 87BE107**  
 BEE M., FOULON M., AMOUREUX J.P., CAUCHETEUX C., POINSIGNON C.:  
 The glassy phase of 1-cyanoadamantane; A study of the molecular reorientations by high-resolution quasi-elastic neutron scattering.  
 JOURNAL OF PHYSICS C VOL.20, PP. 337-349 (1987)
- 87GE108**  
 GELLETLY W., LARYSZ J.R., BOERNER H.G., CASTEN R.F., DAVIDSON W.F., MAMPE W., SCHRECKENBACH K., WARNER D.D.:  
 The  $^{173}Yb(n,\gamma)^{174}Yb$  reaction and the level scheme of  $^{174}Yb$ .  
 JOURNAL OF PHYSICS G VOL. 13, PP. 69-92 (1987)
- 87SC109**  
 SCHRECKENBACH K., SAWICKI J.A., MAIER-KOMOR P., KALVIUS G.M., WAGNER F.E.:  
 Contact electron density in Au-alloys. HYPERFINE INTERACTIONS VOL 28, PP. 701-704 (1986)
- 87TO110**  
 TORBET J., TIMMINS P.A., LVOV Y.:  
 Packaging of DNA in cauliflower mosaic virus and bacteriophage  $\Phi$  studied with magnetic birefringence.  
 VIROLOGY VOL. 155, PP. 721-725 (1986)
- 87HE111**  
 HEIDEMANN A., ECKERT J., PASSELL L., HAEUSLER W.:  
 Pressure dependence of tunneling and librational modes of coupled methyl groups in lithium acetate.  
 ZEITSCHRIFT FUER PHYSIK B VOL. 66, PP. 75-81 (1987)
- 87GA112**  
 GALLOIS B., GAULTIER J., BECHTEL F., FILHOL A., VETTIER C.:  
 Low temperature and high pressure (1.7 K and  $7 \times 10^2$  MPa) structure of the first superconducting organic salt (TMTSF) $_2$ PF $_6$ .  
 MOLECULAR CRYSTALS AND LIQUID CRYSTALS BULLETIN VOL. 1, PP. 259-260 (1986) MOLECULAR CRYSTALS AND LIQUID CRYSTALS VOL. 148, PP. 279-293 (1987)
- 87BE113**  
 BEWERSDORFF H.W., FRINGS B., LINDNER P., OBERTHUER R.C.:  
 The conformation of drag reducing micelles from small-angle-neutron-scattering experiments. RHEOLOGICA ACTA VOL. 25, PP. 642-646 (1986)
- 87FE114**  
 FEREY G., PAPE R., DE, LEBLANC M., PANNETIER J.:  
 Ordered magnetic frustration: VIII. Crystal and magnetic structures of the pyrochlore form of FeF $_3$  between 2.5 and 25 K from powder neutron diffraction. Comparison with the other varieties of FeF $_3$ .  
 REVUE DE CHIMIE MINERALE VOL. 23, PP. 474-484 (1986)
- 87SC115**  
 SCHULTZ A.J., WANG H.H., WILLIAMS J.M., FILHOL A.:  
 The structure of  $\beta^*(BEDTTF)_2I_3$  at 4.5 K and 1.5 Kbar.  
 PHYSICA B VOL. 143, PP. 354-356 (1986)  
 (PROCEEDINGS OF THE XV YAMADA CONFERENCE, YAMANASHI, JAPAN, MAY 26-30, 1986)
- 87VE116**  
 VETTIER C., BURLET P., ROSSAT-MIGNOD J.:  
 Neutron scattering studies of anomalous rare earth compounds.  
 JOURNAL OF MAGNETISM AND MAGNETIC MATERIALS VOL. 63 & 64, PP. 18-24 (1987).  
 (PROCEEDINGS OF THE ICAREA, GRENOBLE, JULY 7-11, 1986)
- 87CH117**  
 CHATTOPADHYAY T., BURLET P., ROSSAT-MIGNOD J., BARTHOLIN H., VETTIER C., VOGT O.:  
 High pressure neutron and magnetization investigations of the magnetic ordering in CeSb.  
 JOURNAL OF MAGNETISM AND MAGNETIC MATERIALS VOL. 63 & 64, PP. 52-54 (1987). (PROCEEDINGS OF THE ICAREA, GRENOBLE, JULY 7-11, 1986)
- 87OS118**  
 OSBORN R., LOEWENHAUPT M., RAINFORD B.D., STIRLING W.G.:  
 Magnons in CeAl $_2$ .  
 JOURNAL OF MAGNETISM AND MAGNETIC MATERIALS VOL. 63 & 64, PP. 70-72 (1987) (PROCEEDINGS OF THE ICAREA, GRENOBLE, JULY 7-11, 1986)
- 87LO119**  
 LOEWENHAUPT M., REICHARDT W., PYNN R., LINDLEY E.:  
 The unusual excitation spectrum of CeAl $_2$ .  
 JOURNAL OF MAGNETISM AND MAGNETIC MATERIALS VOL. 63 & 64, PP. 73-75 (1987). (PROCEEDINGS OF THE ICAREA, GRENOBLE, JULY 7-11, 1986)
- 87KN120**  
 KNOPP G., SPILLE H., LOIDL A., KNORR K., RAUCHSCHWALBE U., FELTEN R., WEBER G., STEGLICH F., MURANI A.P.:  
 An inelastic neutron scattering study on the heavy fermion systems CeCu $_2$ Ge $_2$  and CeAg $_2$ Ge $_2$ .  
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	87BL152		87GA1048		87LA131
	87KE240	DORNER B.	87BU175		87LA168
COLLETTE C.	87BO1064		87DO192		87LA213
COLLIN G.	87RE292		87KA273		87LA397
COLOMBAN P.	87CO1008		87IV304	FERGUSON M. MCL.	87WA398
COLOMBET P.	87CA1049		87DO432	FETTERS L.J.	87HU328
COLVIN G.G.	87CI101	DOSCH H.	87DO192		87HU329
	87CO144	DOUCHIN F.	87KO10T	FIGLARZ M.	87DA1021
	87CA145	DRIESSEN G.	87RI219	FILHOL A.	87FI19T
	87CO177	DROUET L.	87BA400		87AN20T
	87SU232	DUBBERS D.	87MU211		87AN22T
	87GE245		87BI261		87GA112
	87SU318	DUBOIS J.M.	87JA184		87SC115
	87RE292		87PA208		87GA147
COMES R.	87PO315		87DU286	FINNEY J.L.	87KU227
CONARD J.	87CO236		87DU340		87SA228
CONVERT P.	87BR242		87DU346	FISHMAN S.	87FI254
	87BO363		87JA386	FITCH A.N.	87JO317
	87CO1047	DUCASSE L.	87GA147		87KE406
COPPOLA R.	87CR189	DUNN M.	87DO225		87WR413
COQUET E.	87CO270	DUPLESSIX R.	87RA173	FJELLVAG H.	87LE193
	87ZU253		87CA1017	FLOTTMANN T.	87FL294
CORTI M.	87CO1005	DUPUY J.	87EL226		87FL355
COSGROVE T.	87CO1065		87DU263	FORGAN E.M.	87GI231
COULOMB J.P.	87KR1002		87JA359	FOULON M.	87BE107
	87BI1016		87MA360		87BE1010
COULON G.	87CO278		87CA411	FOURNIER J.M.	87CA337
COWLAM N.	87FE1015		87EL414	FRANK A.	87JO293
COWLEY R.A.	87HI170	DURAND D.	87AN171	FRANK V.L.P.	87FR332
	87GR1051	EBEL J.P.	87PO420	FRANSE J.J.M.	87FR214
CRETTEZ J.M.	87CR189	ECKERT J.	87HE11T	FREILAENDER P.	87FR396
	87CO270		87HE111		87VA1059
CROWLEY T.L.	87CO1065		87VO1004		87VA1068
CUILLEL M.	87CU427	ECKOLD G.	87BE105	FREIMUTH H.	87FR319
CUMMINGS S.	87EN330		87BE320		87WI331
CUMMINS P.G.	87CU1042		87RA321		87SC333
CURRAT R.	87MO148		87RA322		87SC334
	87AN171		87KU323	FRELTOFT T.	87FR287
	87PI255		87SE1052	FREYLAND W.	87VA1059
	87SI341	EDWARDS D.M.	87RA127	FREYSSINET J.M.	87TO384
	87LI437	EGIDY T. VON	87WH103	FRINGS B.	87BE113
CYROT-LACKMANN F.	87CH436		87BO279	FRINGS P.H.	87FR123
D'HONDT P.	87WA234		87BA312		87FR214
DAMAY P.	87LE154		87WI313		87SI426
	87LE358		87KR374	FRIT B.	87LA356
DANIEL M.F.	87DA1021	EHRHART P.	87UR401	FRUCHART D.	87SO217
DANOT M.	87CA1049	EICHHORN G.	87KU323		87CH296
DAVIDSON W.F.	87WH103	EISENBERG H.	87CA266	FRUCHART R.	87CH296
	87GE108	ELARBY-AOUIZERAT A.	87EL226		87DU346
	87BA312		87DU263	FUJARA F.	87FU301
DAVIES H.A.	87FE1015		87EL414	GAEHLER R.	87BA178
DEDEK U.	87SC366	ELGADI M.	87BO363	GALERA R.M.	87LA121
DEGENHARDT D.	87DE335	ENCISO E.	87BE434		87GA128
DEGIORGIO V.	87ZU253	ENDERBY J.E.	87BA207		87LA257
DEGHAN K.	87DU286		87EN259	GALLET F.	87NO132
DELAPALME A.	87LA385		87EN330		87GA336
DELUGEARD Y.	87LA157		87EN347	GALLIER J.	87TO160
DENOYER F.	87AN171		87EN348	GALLOIS B.	87GA112
	87SI341		87EN349		87GA147
DEPORTES J.	87DE235		87BA370	GARAWI M.	87GA1048
DERDOUR S.	87SO217	ER RAKHO L.	87EN380	GARCIA N.	87BE434
DESBAT B.	87DA1021	ERKELENS W.A.C.	87MI215	GARDNER P.P.	87FE1015
DESRE P.	87YA364		87RE176	GASPARD J.P.	87GA430

GAULTIER J.	87GA112		87HA299		87HU382
	87GA147		87SU318	HUBER K.	87HU209
GAUNE-ESCARD M.	87BR174	HARTMANN O.	87HA353		87HU328
GAVARRI J.R.	87LU139		87HA354		87HU329
	87CH181	HASSE R.W.	87ST142	HUNGERFORD P.	87SU232
	287CA1049	HAYES W.	87CL272		87BO279
GEICK R.	87RA321	HAYTER J.B.	87CH252		87BA312
	87RA322		87CL272	HUTCHINGS M.T.	87BU272
	87KU323		87CU1042		87HU1003
	87SI426		87BU1044		87HU1031
	87GR1051		87BU1045	IFF-KFA	87IL8T
GELLETLY W.	87GE108	HEATH T.G.	87CO1005	IHLE G.	87IH390
	87GE245		87CO1065	ILL	87IL8T
GELTENBORT P.	87CO177	HEGER G.	87RA322	ILLINI T.	87DO432
	87KO244		87LA397	INGRAM B.T.	87BU1044
GEORGE B.	87JA182		87GR1051		87BU1045
	87JA306	HEIDEMANN A.	87HE11T	IVANOV A.S.	87IV304
GERAND B.	87DA1021		87PR15T	JACKSON I.P.	87JA1006
GERING E.	87RE1030		87HE111		87JA1066
GEURTS J.	87KU323		87HE150	JACOBE J.	87BU104
GEYER A. DE	87GE381		87GR153	JACOBS L.	87JA238
	87TA389		87PE169	JAL J.F.	87EL226
GHOSH R.	87GH135		87HE212		87DU263
GIBAUD A.	87GI291		87MU362		87JA359
GIBBONS E.P.	87GI231	HEIMING A.	87FL355		87MA360
GIEGE R.	87PO420	HEITJANS P.	87FR396		87CA411
GIGNOUX C.	87GI412		87VA1059	JANOT C.	87JA182
GIGNOUX D.	87GI373		87VA1068		87JA183
GILBERD P.W.	87BE1026	HEMPELMANN R.	87HE180		87JA184
GILLON B.	87BO206		87RI219		87PA208
	87BO316		87HE222		87JA306
	87LA157		87HA353		87JA307
GIRARD A.	87BE435		87HA354		87DU340
GIRAUD P.	87MO196	HENDRIKX Y.	87HE371		87DU346
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GOEBEL H.	87GO13T	HERDMAN G.J.	87EN330	JOBIC H.	87JO162
	87GO16T	HERTZ H.G.	87BE167		87JO317
GOENNENWEIN F.	87KO244	HERVIEU M.	87MI215		87JO1025
GOFFART J.	87GA430	HERZ J.	87BO1064	JOHNSON S.M.	87JO250
GOLDMAN A.M.	87JA238	HESS S.	87LI18T	JOHNSON W.L.	87MA143
GOLDSTONE J.A.	87VO1004	HEWAT A.W.	87HE9T	JOLIE J.	87JO293
GOLUB R.	87KI368		87BR174		87SE408
GOMPF F.	87RE1030		87CH181		87SE409
GONZALEZ-CALBET J.	87RO241		87CA239	JOSE J.V.	87CH102
GRAEFENSTEDT M.	87GR281		87HE308		87JA238
GREEN R.M.	87HO1036		87HE326	JOUBERT J.C.	87TU185
GREMPEL D.R.	87FI254	HEWAT E.A.	87HE410	JUNG S.	87MU362
	87GR267		87HE308	JUST W.	87BR325
GRENIER J.C.	87RO241	HEYDE K.	87HE410	KADONO R.	87KA352
GRIEB T.	87GR1051		87JO293	KAHN R.	87PO420
GRIMM H.	87MO159		87SE408	KAKURAI K.	87ST204
	87MA205		87SE409		87KA273
	87SE1052	HEYN M.P.	87SE1058	KALUS J.	87BA178
	87BU17T	HICTER P.	87MA143		87HO1055
	87GR153		87MA365		87KA1056
GRONEMEYER C.	87MO196	HIGGINS S.A.	87HI170		87HO1057
	87MO361		87GR1051	KALVIUS G.M.	87SC109
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GUENET J.M.	87GU1054	HLAWATSCH G.	87WI313	KAPPLER J.P.	87BA312
GUENTHERODT H.J.	87SU1028	HODEAU J.L.	87BO125	KARLSSON E.	87LA257
	87SU1062	HOFF R.W.	87WH103		87HA353
	87GU158	HOFFMANN H.	87HO1055	KEARLEY G.J.	87HA354
GUILLAUME F.	87EN347		87KA1056		87KE137
GULLIDGE P.M.N.	87HA354		87HO1057		87KE151
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HAENSEL R.	87SC333	HONG M.	87HO1063		87KE377
	87SC334	HOROVITZ B.	87MA205		87KE406
	87DE335		87HO141		87BL433
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	87PR1012	HORSEWILL A.J.	87HO1036	KEMMLER-SACK S.	87BR325
	87PR1014	HOWARD J.A.K.	87LO1050	KEMPTON J.	87HA354
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HAHN T.	87BE105		87CO144	KERR S.A.	87CI101
	87BE320		87CO177		87CO177
HAMADA S.A.	87HA299		87KE195		87KE195
HAMELIN B.	87BU104		87HA299		87BO279
HAMILTON W.A.	87HA247		87WI313	KEYSER U.	87GR281
HAMILTON W.D.	87SU232	HUANG J.S.	87HU344	KIEMEL R.	87BR325

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	87ME424		87UR401		87MA290
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	87LA393	LAVIRON C.	87TU185		87MA365
	87LA394	LE BAIL A.	87GI291	MAREZIO M.	87BO125
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KNORR K.	87KN120		87WA161		87HE410
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KOLLHOFF H.	87KN163	LECHNER M.D.	87VE277	MARUANI J.	87BO206
	87LA394	LECHNER R.E.	87TO160		87BO316
KOLLMAR A.	87KO10T	LECLERCQ F.	87LE154	MATHIEU C.	87JA359
KONDUROV I.A.	87BO279		87LE358		87MA360
KOSSAKOWSKI R.	87BU17T	LEFEBVRE J.M.	87CO278	MATSUZAKI T.	87KA352
KOSSLER W.J.	87HA354	LEGRAND Y.	87BA400	MATTENBERGER K.	87LA385
KOSTORZ G.	87MU298	LEHMANN M.S.	87LE193	MAY R.P.	87PO376
	87CE303		87KU224		87SP407
KOUZES R.T.	87WH103		87KU372	MAYNARD R.	87WO422
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KRATZ K.L.	87OH283		87RA322		87AK429
KREXNER G.	87PI255		87KU323	MCEWEN K.A.	87JE191
KRILL G.	87BO125	LEIDERER P.	87FR332		87GI231
KRIM J.	87KR1002	LEJAY P.	87CA239		87ST417
KRUMPOLC M.	87KN311	LEQUIEN S.	87LE201	MCINTYRE G.J.	87MO140
KRUSCHE B.	87W1313	LETOFFE J.M.	87DU263		87BA284
	87KR374		87CA411		87RE285
	87KR375		87EL414		87RE292
KRUSE J.	87KR399	LEVY F.	87MO148		87TU383
KUHS W.F.	87KU224	LEWIT-BENTLEY A.	87BE419	MCKERGOW M.W.	87MC197
	87KU227		87PO420	MCMAHON T.D.	87KE195
	87NE256	LEYTE J.C.	87BA370	MCMULLAN R.K.	87MO140
	87KU323	HERSONNEAU G.	87OH283	MCWHAN D.B.	87MA205
	87CA337	LI W.H.	87LI203	MENOVSKY A.A.	87FR214
	87KU372	LIAUD P.	87BU17T	MERCIER R.	87MA248
KULLMANN W.	87RA322		87BU104	MEYER R.A.	87ME282
	87KU323	LIEB K.P.	87W1313	MEZEI F.	87ME138
KUMAR K.	87SU232		87KR374		87ME424
KURODA K.	87BU104		87KR375		87VO1004
KVENTSEL G.F.	87PA265	LILJAS L.	87BE419	MICHALOWICZ A.	87BU104
KWO J.	87MA205	LINDLEY E.	87LO119	MICHAUDON A.	87MI378
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	87LA213	LINDNER P.	87LI18T	MICHAUX B.	87CR189
	87LA324		87BE113	MICHEL C.	87MI215
	87LA403	LIPPERT W.	87RA172	MICNAS R.	87MI124
LAHIOUEL R.	87LA121	LIU D.W.	87LI437		87RO268
	87LA122	LOEWENHAUPT M.	87OS118		87MI416
	87LA257		87LO119	MIEKELEY W.	87YO402
LALIGANT Y.	87LA131		87WA1039	MIKOU A.	87LA356
	87LA397		87HO1063	MILNER S.T.	87HU344
LAMCHARFI T.	87GA147	LOGINOV YU.E.	87BO279		87HU382
LAMLOUMI J.	87LA405	LOIDL A.	87KN120	MIREBEAU I.	87LE201
LAMPARTER P.	87MU1001	LONGUEVILLE W.	87BE1011	MITROFANOV N.L.	87IV304
LANDER G.H.	87LA274		87LO1032	MONCEAU P.	87MO148
	87LA275	LOONG C.K.	87HO1063	MONKENBUSCH M.	87MO159
	87LA276	LORBER B.	87PO420		87MO327
	87CA337	LOUCA P.	87LO1050	MONNAND E.	87ME282
	87LA385	LOUGHEED R.W.	87WH103	MONS J.	87LA157
LANGEL W.	87KN163	LOWDE R.D.	87HU1003	MOORE M.S.	87KO244
	87LA164	LUCAS B.W.	87BY1046	MORAS D.	87PO420
	87PR165	LUCAZEAU G.	87LU139	MORKEL C.	87MO196
	87HO190		87GU158		87MO361
	87LA393	LVOV Y.	87TO110	MORONEY L.M.	87BA1020
	87LA394	MACDONALD J.E.	87CL272	MOSS B.K.	87MO140
LANIER R.G.	87WH103	MAGERL A.	87CA149	MOUSSA F.	87BE105
LANKFORD W.F.	87HA354		87W1156		87LA157
LAPP A.	87BO1064		87CA166		87AN171
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LARTIGUE C.	87KO10T		87MA218	MUELLER P.	87MU362
	87LA404		87W1220	MUELLER P.P.	87MU298
	87LA405		87ST233	MUENNICH F.	87GR281
LARYSZ J.R.	87GE108		87CL272	MUENZEL J.	87RA172
LASSEGUES J.C.	87CO1008		87W1367	MURANI A.P.	87KN120
	87DA1021		87FR396		87LA121
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MUTIN J.C.	87CR189	PAPE R.DE	87FE114	RABELL V.	87RA172
MUTTERER M.	87KO244	PARK P.	87SU232	RAINFORD B.D.	87OS118
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NAUMANN E.	87GR1051	PASTUREL A.	87MA290	RANNINGER J.	87MI124
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NEILSON G.W.	87EN330	PAVUNA D.	87CH436		87RA415
NELMES R.J.	87NE256	PAWLOWSKA Z.	87PA265		87MI416
	87RE285	PEARSON D.	87AL305	RAUCH H.	87RA1007
	87TU383	PEISL J.	87BU175		87RA1067
NEMOZ C.	87BU104		87DO192	RAUCHSCHWALBE U.	87KN120
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	87CA246		87CU1042		87CA239
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	87HA353	PERSCHIED B.	87HO190	REES B.	87PO420
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	87BU1038	PETRY W.	87TO160	REINHARD L.	87MU298
	87LA1043		J87PE169	RENARD J.P.	87RE176
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NOVOTNY V.	87KN311		87FU301		87RE1030
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	87NO133		87SC366		87JO317
	87UW134		87WA398		87JO1025
	87NO314		87YO402	RESTORI R.	87RE285
	87GA336	PFEIFFER B.	87ME282	RICHARD C.	87MU1034
	87NO369		87OH283	RICHARD J.M.	87IH390
	87NO425	PHAM-THI M.	87CO1008		87GI412
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NUGTEREN P.R.	87CA1035	PIERRE J.	87LA121	RICHARDSON R.M.	87WA129
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	87RA271		87GA128	RICHTER D.	87RI155
	87VE277		87LA257		87RE180
	87AL305	PINSTON J.A.	87ME282		87RI219
	87KO244		87BA312		87HE222
OED A.	87OH283	PINTSCHOVĪUS L.	87PI255		87RI230
OHM H.	87BE435	PIRNER H.J.	87IH390		87FR287
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	87HA247	PLUMIER R.	87PL1023		87HU344
OPAT G.I.	87CR189		87PL1041		87RI351
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	87OS229	POINSIGNON C.	87BE107		87HA354
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	87DE235		87SL391	RIETSCHEL H.	87RE1030
OULADDIAF B.	87ME282		87CO1008	RIEUBLAND J.M.	87KN311
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PALMER S.B.	87RO14T	PRAGER M.	87PR15T		87MI416
PANNETIER J.	87FE114		87GR153	ROBERT A.	87RO6T
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# LIST OF ACCEPTED PUBLICATIONS

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Max von Laue (1879-1960)

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Paul Langevin (1872-1946)



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