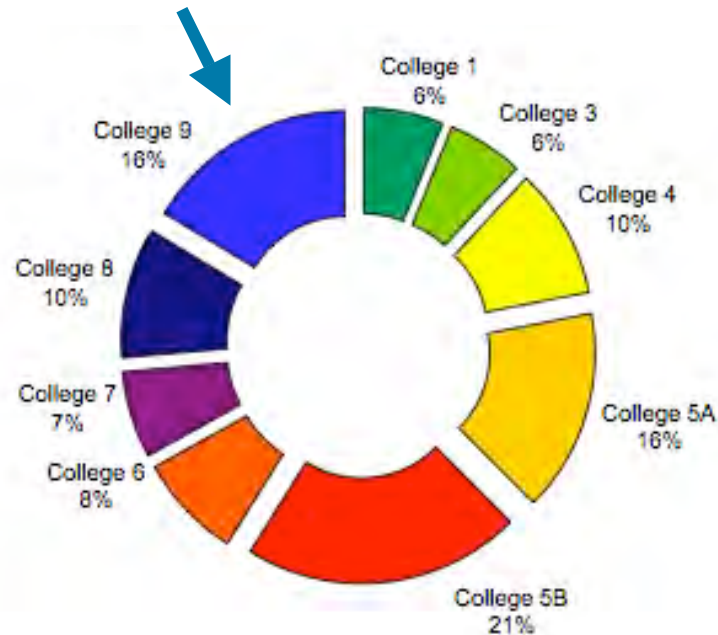


Sample environment in Soft Condensed Matter at the ILL: the PSCM initiative

Giovanna Fragneto



Soft Matter at the

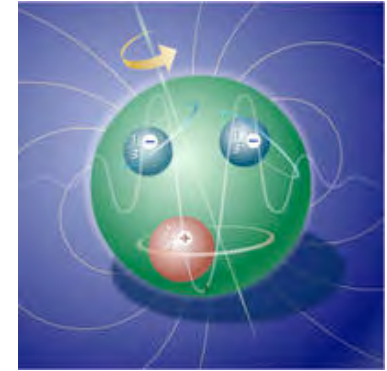


9-10: **Colloidal systems:** micelles, microemulsions, amphiphilic aggregates, surfactant systems, organic and inorganic colloidal particle systems

9-11: **Polymeric systems:** solutions, melts, polyelectrolytes, blends, co-polymers, elastomers, gels, networks, liquid crystal

9-12: **Mixed systems:** organic or inorganic particles with surfactants, polyelectrolytes or polymers, surfactants and polymers,...

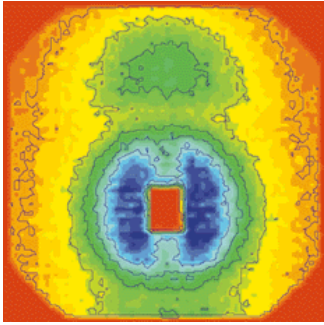
9-13: Soft matter physics with **bio-related molecules**



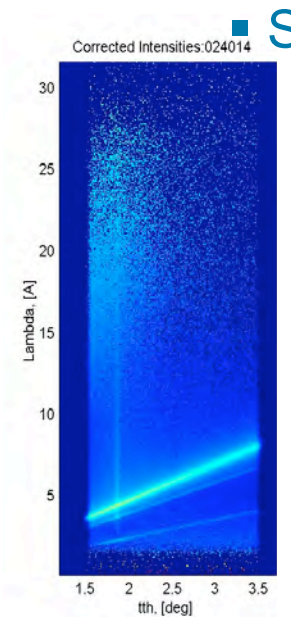
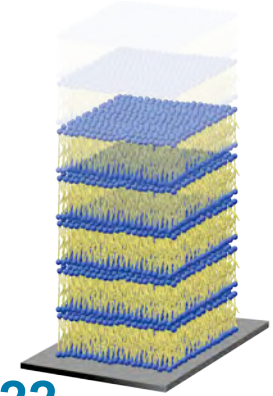
Neutrons strongly scattered by light atoms
Deuterium Labeling & Contrast Variation
Buried systems & Complex sample environment
Non-destructive

Low flux → Big samples

Neutron and synchrotron radiation
techniques present many
complementary aspects and there is a
wide soft matter user community that
benefits from the two



Most requested instruments



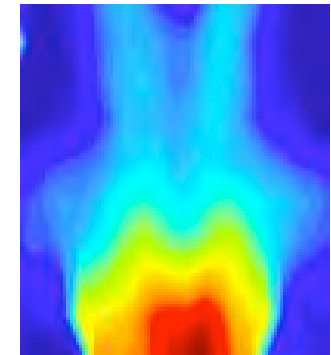
- Small angle neutron scattering diffractometers: **D11** and **D22**

- Reflectometers: **D17** and **ADAM**

- Small momentum transfer diffractometer: **D16**

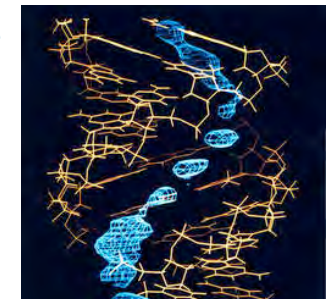
- Time of Flight spectrometers: **IN5** and **IN6**

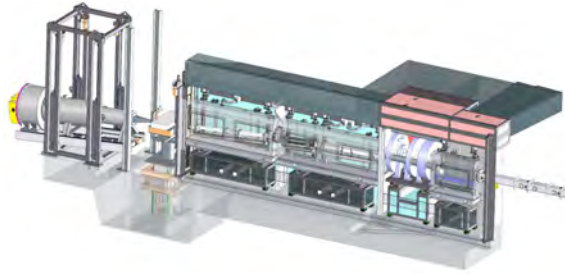
- Spin echo spectrometers: **IN11** and **IN15**



- Cold neutron backscattering spectrometers: **IN10** and **IN16**

- Thermal neutron backscattering spectrometer: **IN13**





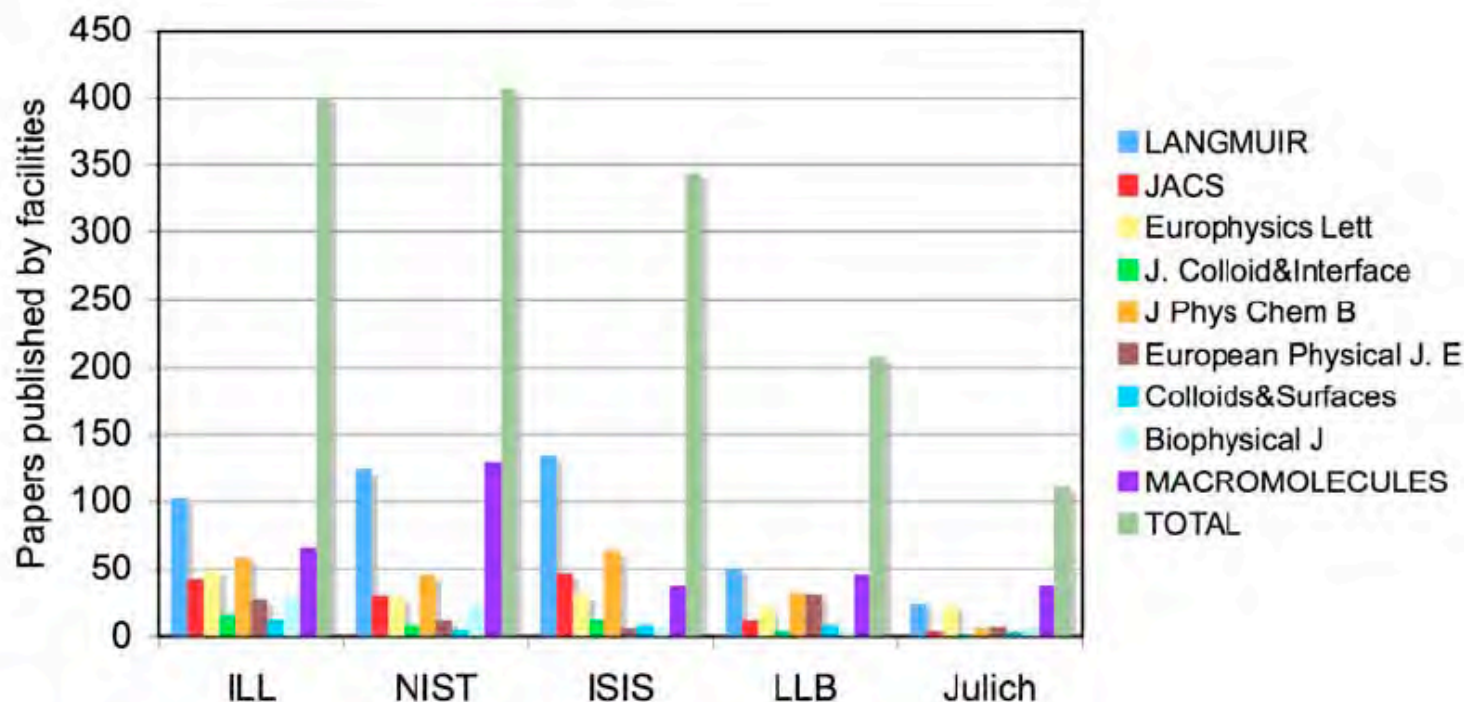
Millennium Programme Projects for instruments (fully or partially) devoted to soft matter

- Cold neutron backscattering spectrometer: **IN16B** (*new*)
 - Time of Flight spectrometer: **IN5** (*upgrade*)
- Reflectometers: **FIGARO** (*new*) **SuperADAM** and **D17** (*upgrade*)
- Small momentum transfer diffractometer: **D16** (*upgrade*)
 - Spin echo spectrometer: **WASP** (*new*)
- Small angle neutron scattering diffractometers: **D11** (*upgrade*) and **D33** (*new*)

a lot of activity that justifies ILL interest in the Partnership for Soft Condensed Matter

Idea comes from ILL and ESRF management
(from need to increase publication output in soft matter)

Period 2000-2006



Why?

- More complex systems (particularly for SANS) - challenging experiments
- Samples prepared on-site and/or not well characterized in advance
- Need complementary information for publishing
- Need in-situ complementary technique
- Not enough members of staff to help with data analysis for new users
-



ILL SOFT MATTER USER MEETING

Institut Laue-Langevin, Grenoble (France)
22 - 24 November 2006
<http://www.ill.fr/softill2006>

Second Announcement

This is the second announcement for an ILL Soft Matter User Meeting organized by the Institut Laue-Langevin.

Soft matter scientists are invited to participate in this meeting and to present their work in the fields of polymers, colloids and interface science, involving both neutrons and complementary techniques. Ample time will be reserved for discussion in order to define the needs of the community concerning an on-site facility for sample preparation and characterization, use of complementary methods and ways to optimize of the use of beam-time.

In view of the possible creation of a Partnership for Soft Condensed Matter on the ILL-ESRF site this workshop will give the unique opportunity to raise user interests concerning structure, scientific orientation and equipment in an early stage.

Meeting chairman: Stefan Egelhaaf

Speakers will include:

Arantxa Arbe (S. Sebastian, S)
Matthias Ballauf (Bayreuth, D)
Paul Bartlett (Bristol, UK)
Bernard Cabane (Paris, F)
Olivier Diat (Grenoble, F)
Julian Eastoe (Bristol, UK)
Stefan Forster (Hamburg, D)
Andrew Glidle (Glasgow, UK)
Regine Von Klitzing (Kiel, D)
Julian Oberdisse (Montpellier, F)
Walter Richtering (Aachen, D)
Tim Salditt (tbc) (Gottingen, D)
Anna Stradner (Fribourg, CH)
Andreas Wischnewski (Juelich, D)

Chairpersons:

Christiane Alba-Simionesco (Paris, F)
Stefan Egelhaaf (Dusseldorf, D)
Eric Geissler (Grenoble, F)
Richard A. L. Jones (Sheffield, UK)
Jean-François Legrand (Strasbourg, F)
Gregory McKenna (Texas, USA)
Dieter Richter (Juelich, D)
Peter Schurtenberger (Fribourg, D)
Robert K. Thomas (tbc) (Oxford, UK)



DEADLINE FOR REGISTRATION (<http://www.ill.fr/softill2006>): **15TH SEPTEMBER 2006**

Advisory Committee:

Christiane Alba-Simionesco, Richard A. L. Jones, Robert L. McGreevy, Dieter Richter, Jens Rieger, Peter Schurtenberger

Local organizing committee:

Trevor Forsyth, Giovanna Fragneto, Bernhard Frick, Isabelle Grillo, Peter Lindner, Peter Timmins, Christian Vettier

Secretary: Karine Sultan

Contact: softill2006@ill.fr



~ 120 participants



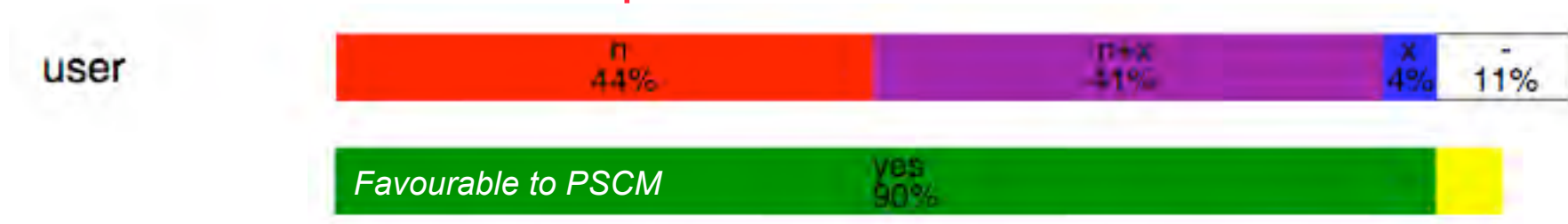
ILL Soft Matter User Meeting
22 - 24 November 2006, Grenoble, France

Chair: Stefan Egelhaaf

Local Organizers: T. Forsyth, G. Fragneto, B. Frick, I. Grillo, P. Lindner, P. A. Timmins, C. Vettier

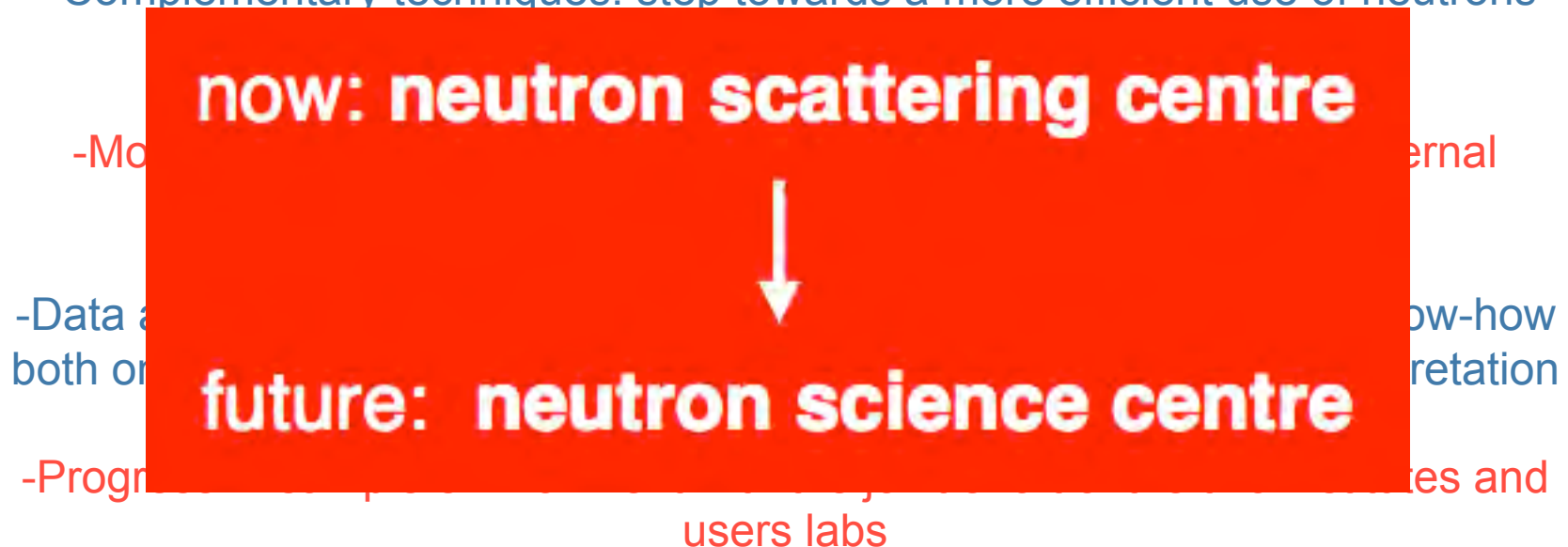
Advisory Committee: C. Alba-Simionesco, R.A.L. Jones, R. McGreevy, D. Richter, J. Rieger, P. Schurtenberger

Questionnaire: Partnership for Soft Condensed Matter



-Sample preparation and characterization

- Complementary techniques: step towards a more efficient use of neutrons



- Complementary equipment would require support by appropriate staffing.

As seen from the ILL, the **main objectives** for the creation of the PSCM are as follows:

- to provide **enhanced support** to soft matter scientists in Europe
- to develop **leadership** in soft matter research with neutron and synchrotron x-rays
- to address **non scattering-expert** communities in the field of soft matter
- to maintain a proper **balance** between service and in-house research

June 2007:

ILL Steering Committee favourable to PSCM and agrees on the creation of the **Soft Matter Lab** in the former biochemistry/deuteriation labs

November 2007:

EMBL agrees to lend lab space in ILL20-2

December 2007:

Start of **ESFRI** grant

December 2007:

Award of **CPER** grant from local bodies for *site renewal* *it includes a building with space for PSCM*

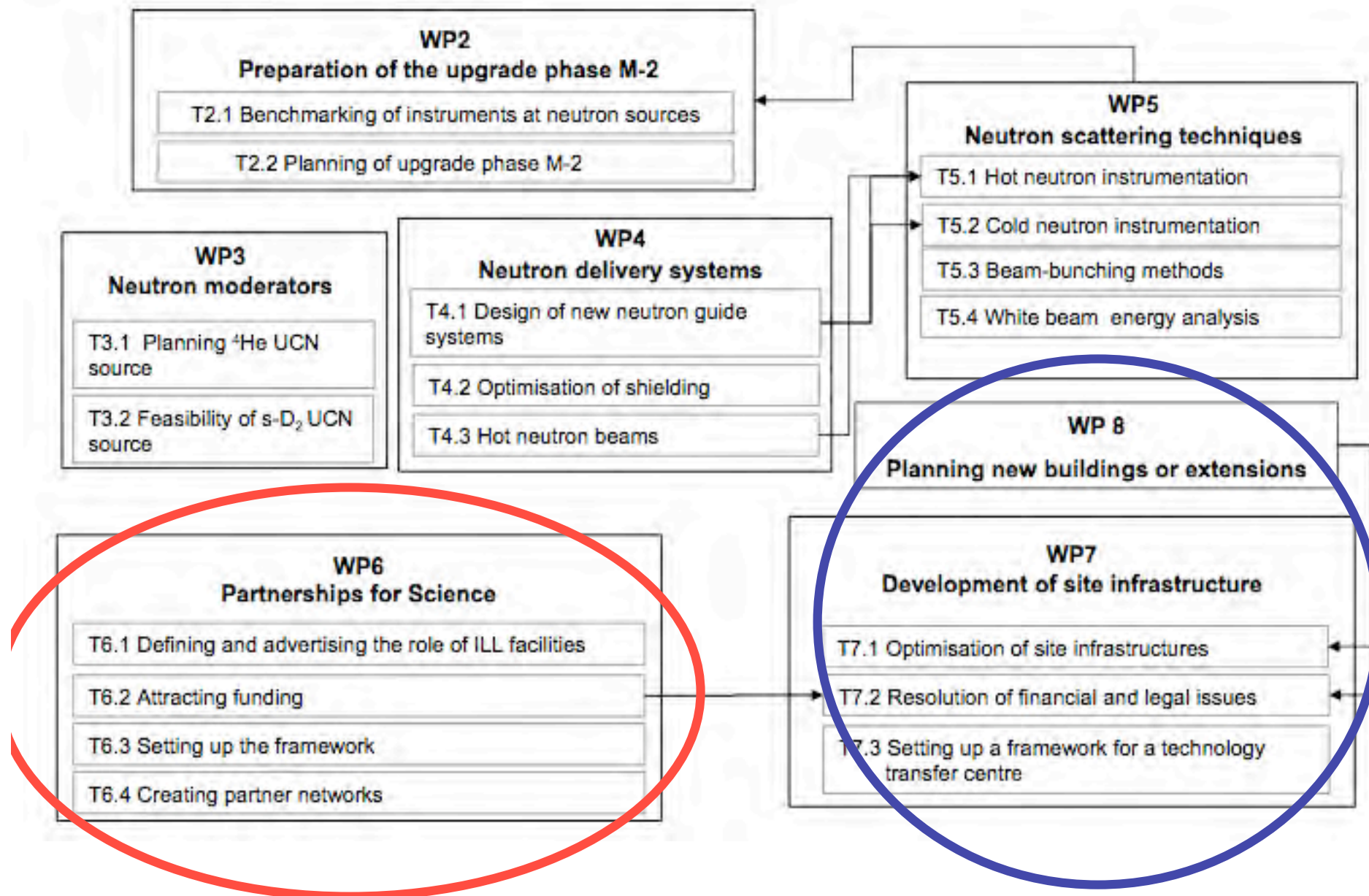
January 2008:

Start of contract ILL Soft Matter Fellow (renewed discussion with ESRF)

ESFRI GRANT

- Prepare for upgrade of ILL
- Improve visibility and research capability of site

B.1.3.2b Flow chart



Tasks in WP6

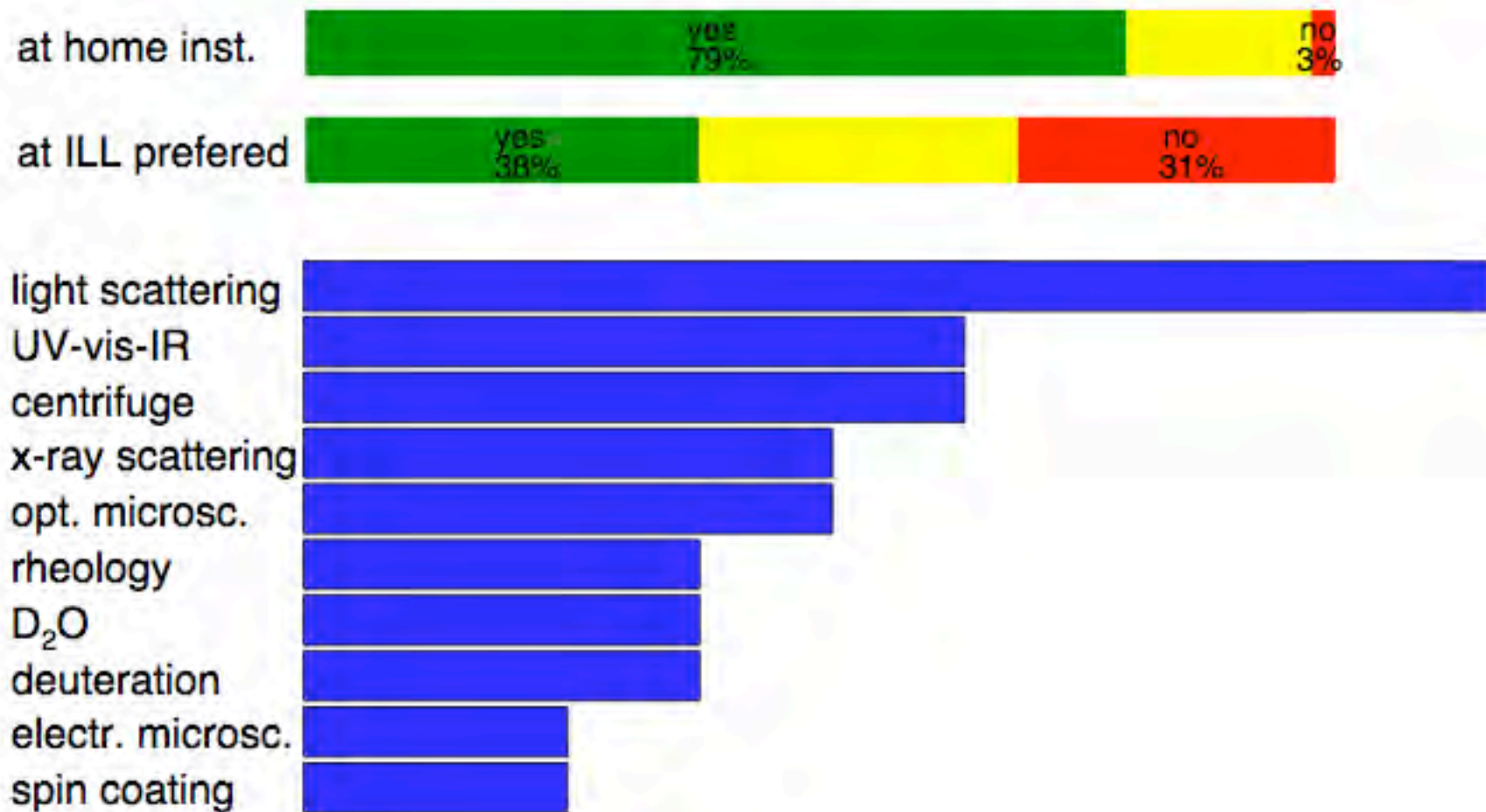
1. Defining and advertising role of ILL facilities
(jan 2008 - dec 2010)
2. Attracting funding and potential partners
(jul 2008 - jul 2009)
3. Setting up the framework
(jul 2008 - dec 2009)
4. Network of Partners
(oct 2008 - dec 2010)



The ILL Soft Matter Laboratories

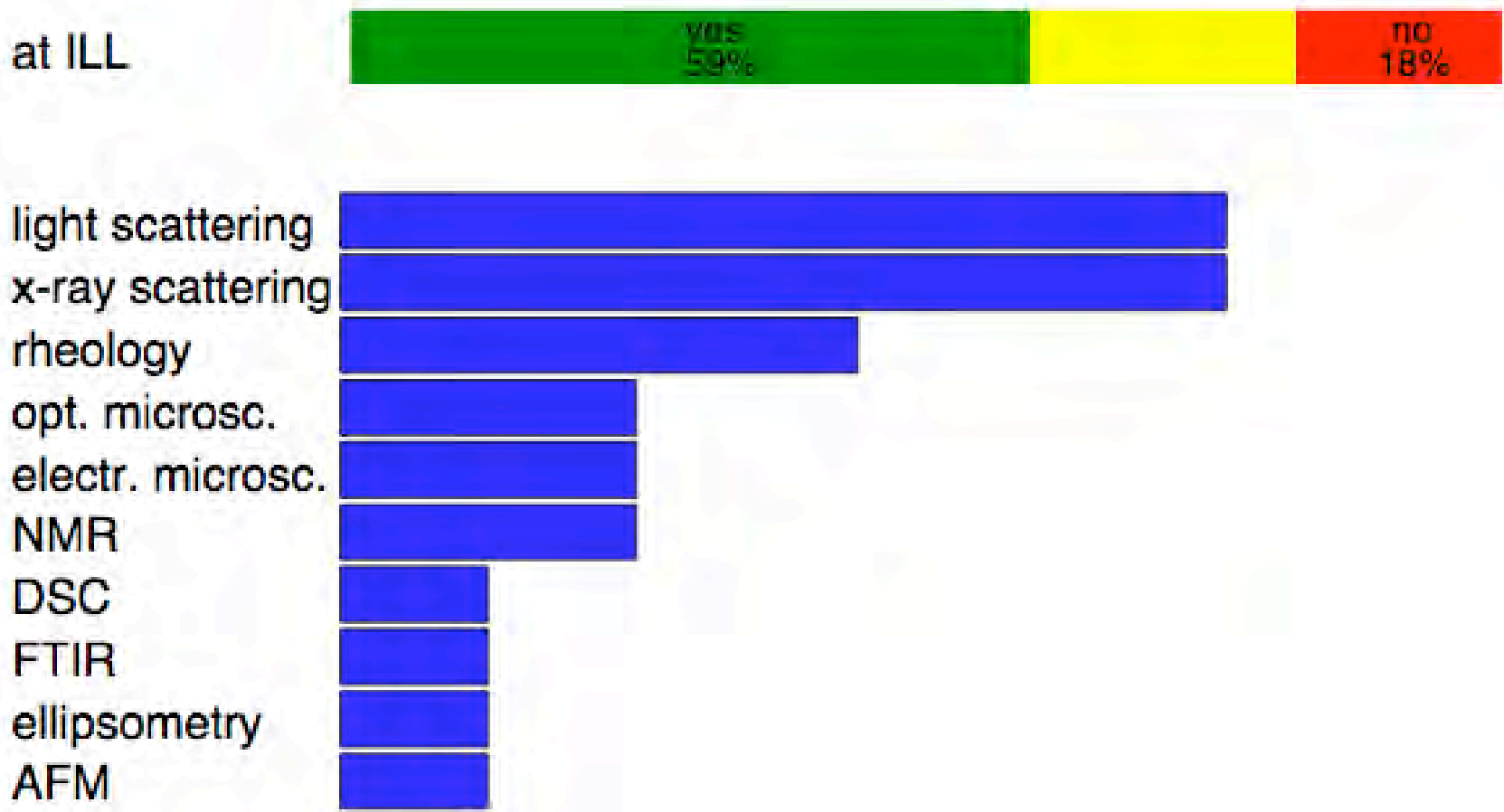
...a “baby” facility for sample preparation
and characterization

Soft Matter User Meeting: what users asked for Sample Preparation and pre-characterization



*From Report on Soft Matter User Meeting
by S. Egelhaaf and P. Schurtenberger*

Soft Matter User Meeting: what users asked as Complementary Techniques



*From Report on Soft Matter User Meeting
by S. Egelhaaf and P. Schurtenberger*



Former Deuteriation Lab



EMBL lending

Spin coating is a procedure used to apply uniform thin films to flat substrates.



Langmuir-Blodgett apparatus for deposition of insoluble layers on surfaces

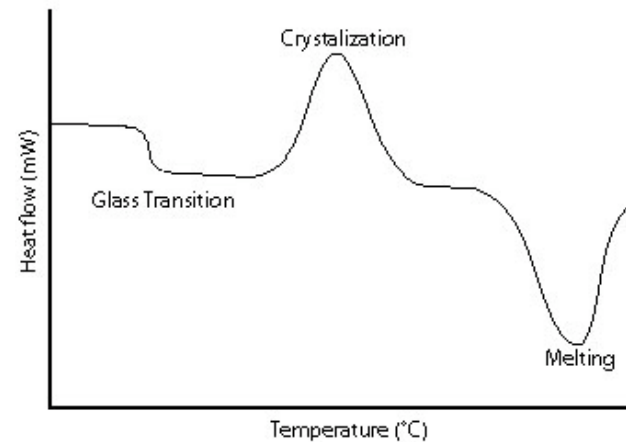


Upon the analysis of the change of polarization of light, which is reflected off a sample, **ellipsometry** can yield information about layers that are thinner than the wavelength of the probing light itself. It is commonly used to characterize film thickness for single layers or complex multilayer stacks ranging from a few angstroms or tenths of a nanometer to several micrometers



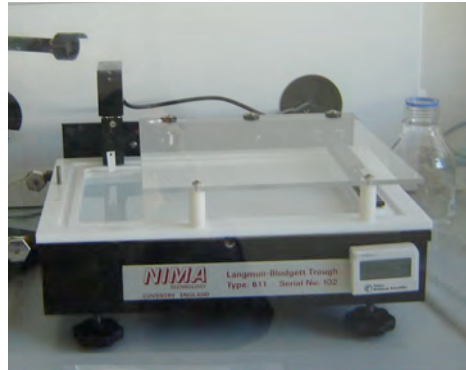


By observing the difference in heat flow between a sample and reference, **differential scanning calorimeters** are able to measure the amount of heat absorbed or released during phase transitions. The ability to determine transition temperatures and enthalpies makes DSC an invaluable tool in producing phase diagrams for various chemical systems

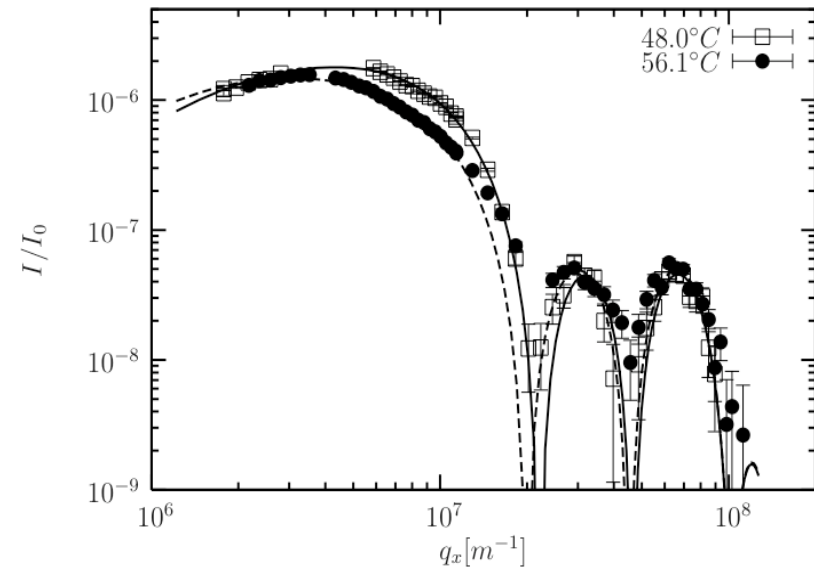
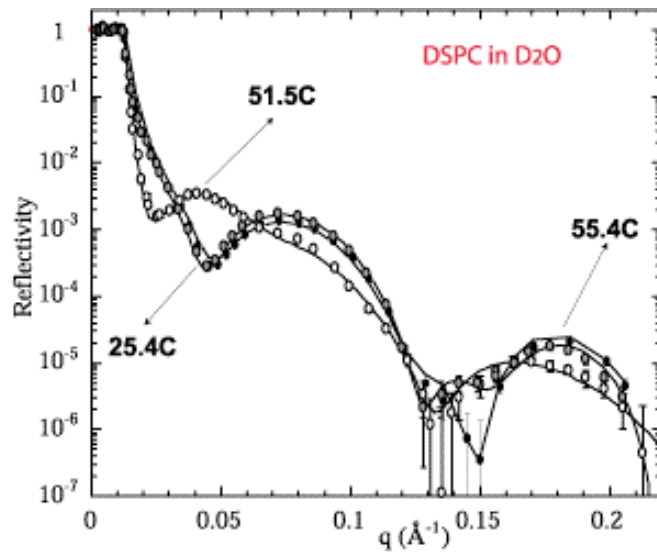
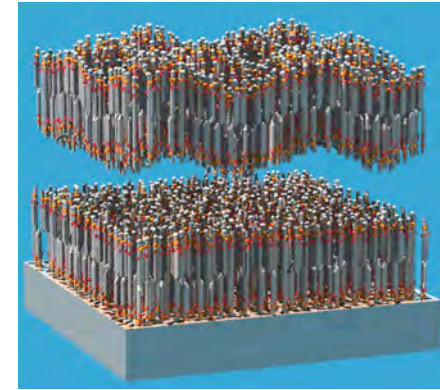


Amphiphilic molecules reduce the surface tension of water by adsorbing at the liquid-gas interface. By measuring the **surface tension** it is possible to quantify the amount of molecules on the surface and verify their purity.

2000-2008



6 ILL PhD thesis
 2 master thesis
 ~40 experiments ILL
 7 experiments ESRF
 2 ISIS 1 NIST
 ~20 publications
 + in prep



$$\langle z(q_{\parallel}) z(q_{\parallel}) \rangle = \frac{k_B T}{A + \gamma q_{\parallel}^2 + \kappa q_{\parallel}^4}$$

Fragneto et al. Europhys Lett 2001, Langmuir 2003

Dailant et al. PNAS 2005

The Partnership should become a “reference” for the soft matter community not only for sample preparation and characterization but also for issues related to “soft” sample environment

Sample environment developments include:

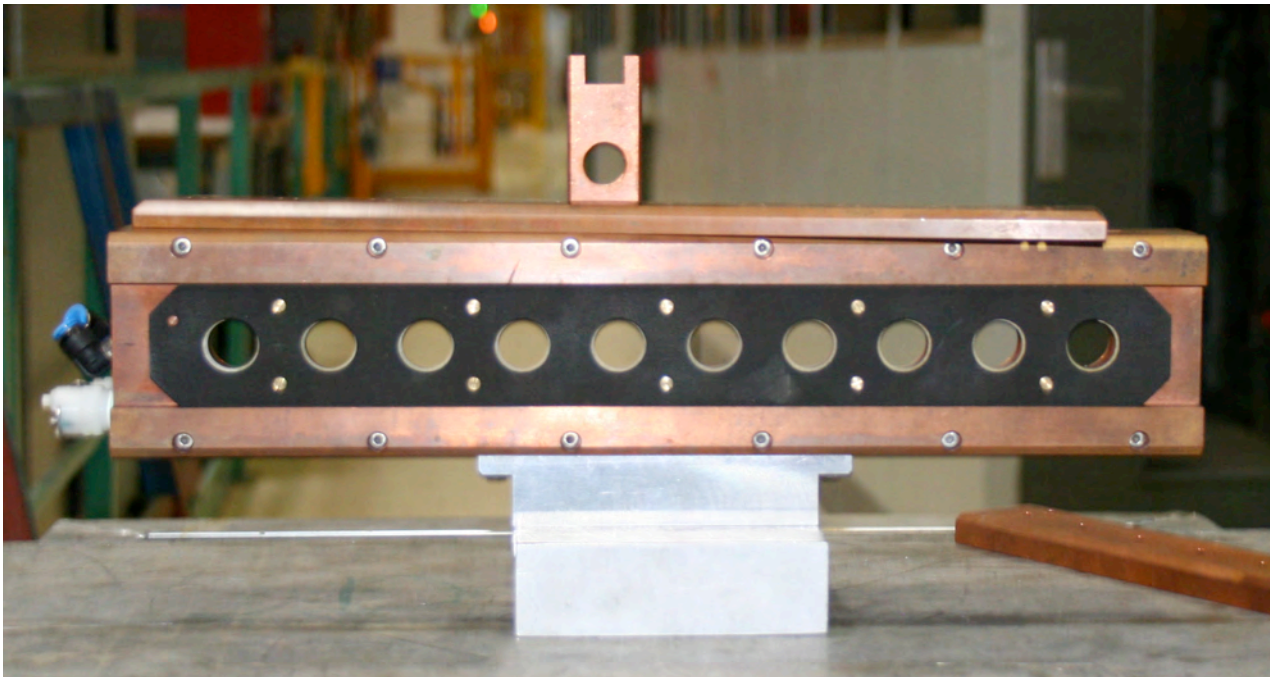
Precise control around room T

T controlled pressure cells

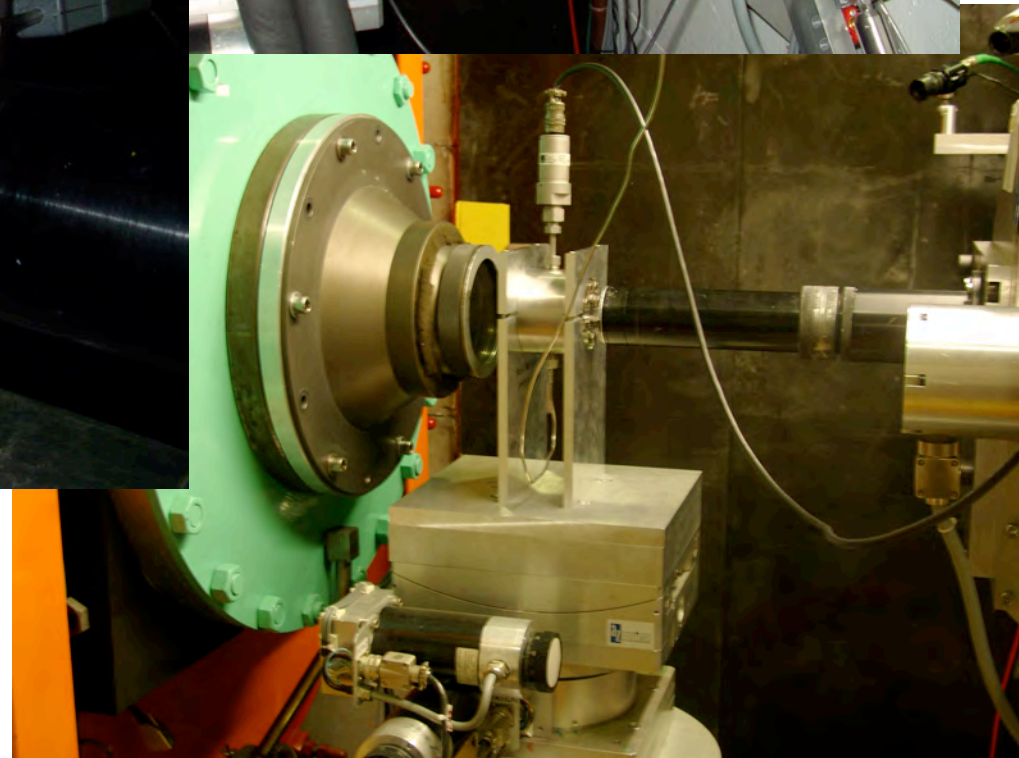
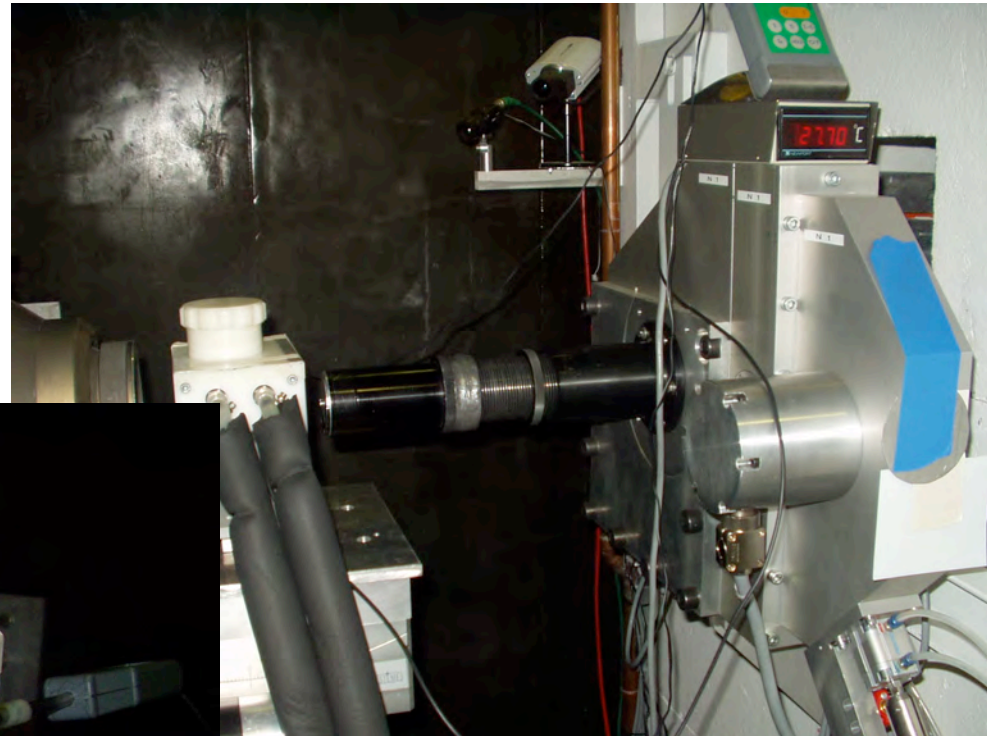
Humidity control (below and above 0°C)

Time-resolved experiments

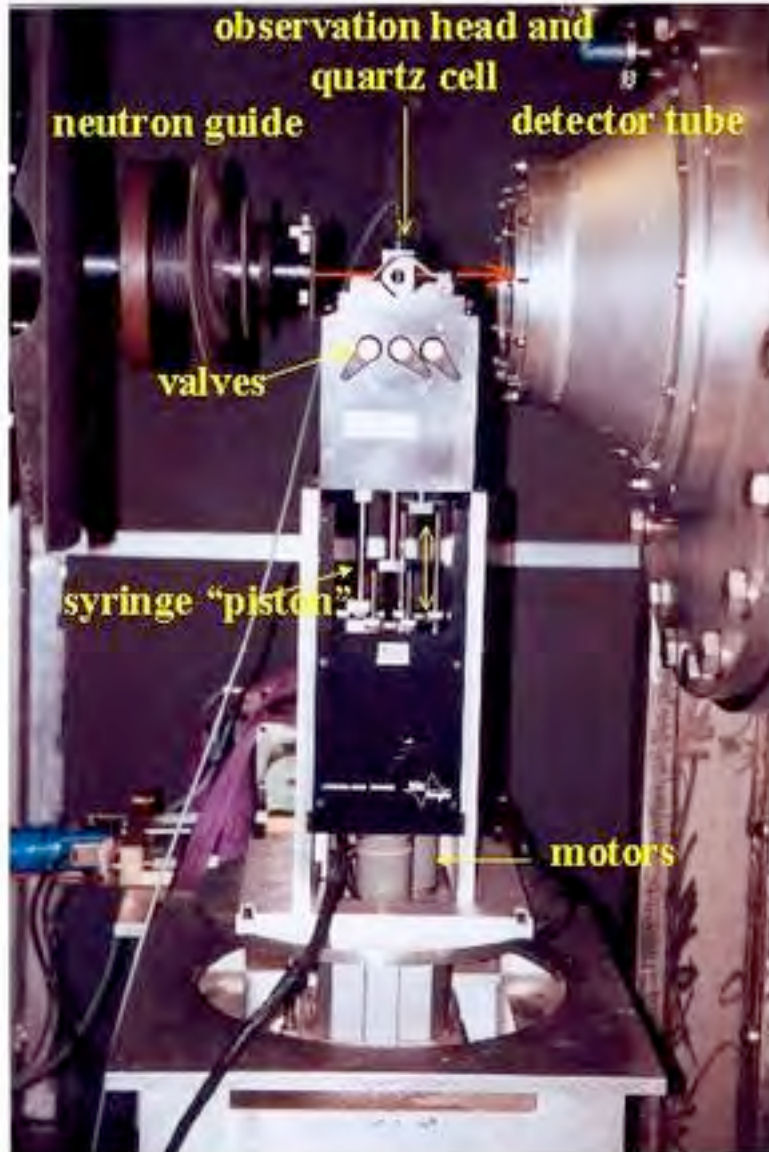
In-situ complementary measurements



D11 pressure cells



D22 stopped flow



D11 Bohlin rheometer



List of additional techniques that could be implemented *immediately* in the Soft Matter Laboratory or as part of sample environment because *expertise is available on-site* as well as the willingness of ILL/ESRF staff to use and maintain the equipment

Funds available for:

Static and Dynamic Light Scattering

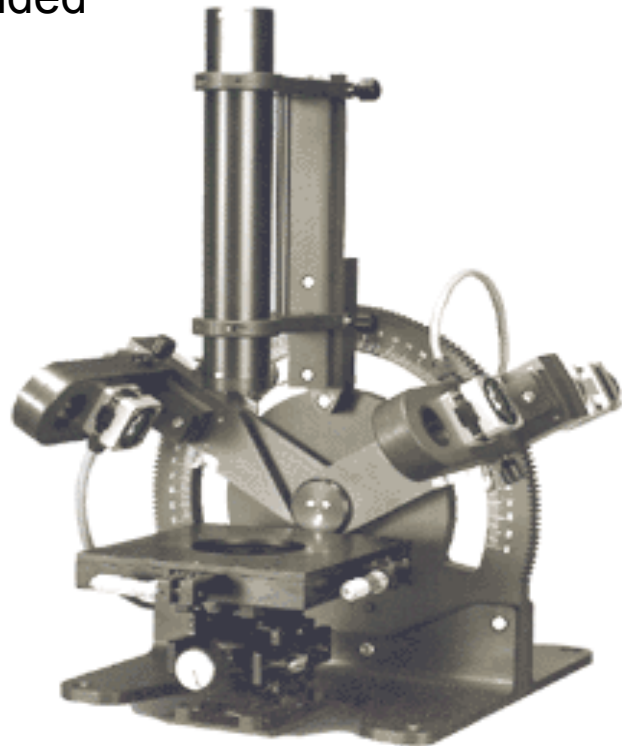
Ralf Schweins

Bruno Demé

Isabelle Grillo

Abdellatif Moussaid/ESRF

ESRF LS set-up to be used for turbid solutions and cases where flexibility is demanded



Ellipsometer for liquid surfaces

Richard Campbell

Giovanna Fragneto

Oleg Konovalov/ESRF

Activities started for:

- Deuterated Lipid Extraction from “waste” products of Deuteriation Lab



PhD Sarah Khanniche

Bruno Demé

Giovanna Fragneto

Philip Callow

Susana Teixeira

Michael Haertlein

On-going discussion for acquisition of:

- X-ray reflectometer

Giovanna Fragneto

Andrew Wildes

(Axel Steuwer, Ken Andersen

Oleg Konovalov ESRF)

Anders Madsen (ESRF)

- SAXS

Franck Gabel (IBS)

(Joe Zaccai, Trevor Forsyth

Isabelle Grillo, Bruno Demé)

- Rheometer

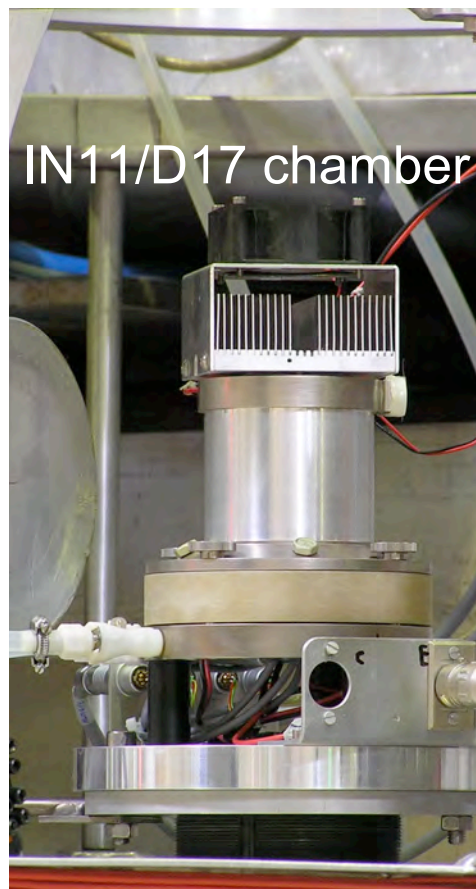
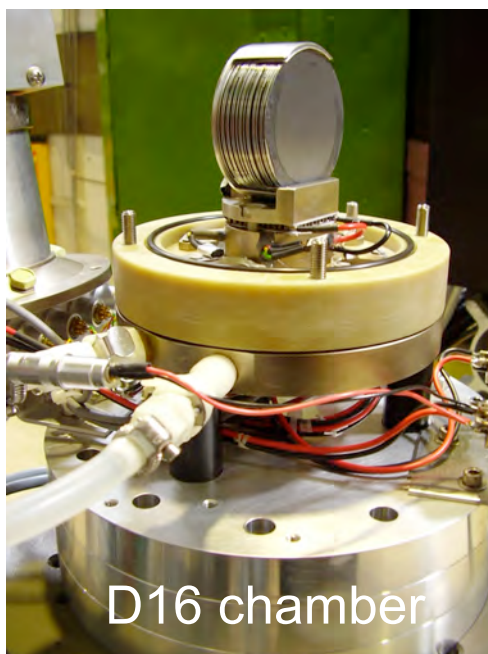
Peter Lindner

(Lionel Porcar, Max Wolff)

Humidity control

Need:

Simultaneous control of T and RH
 Continuous change of RH
 100% RH



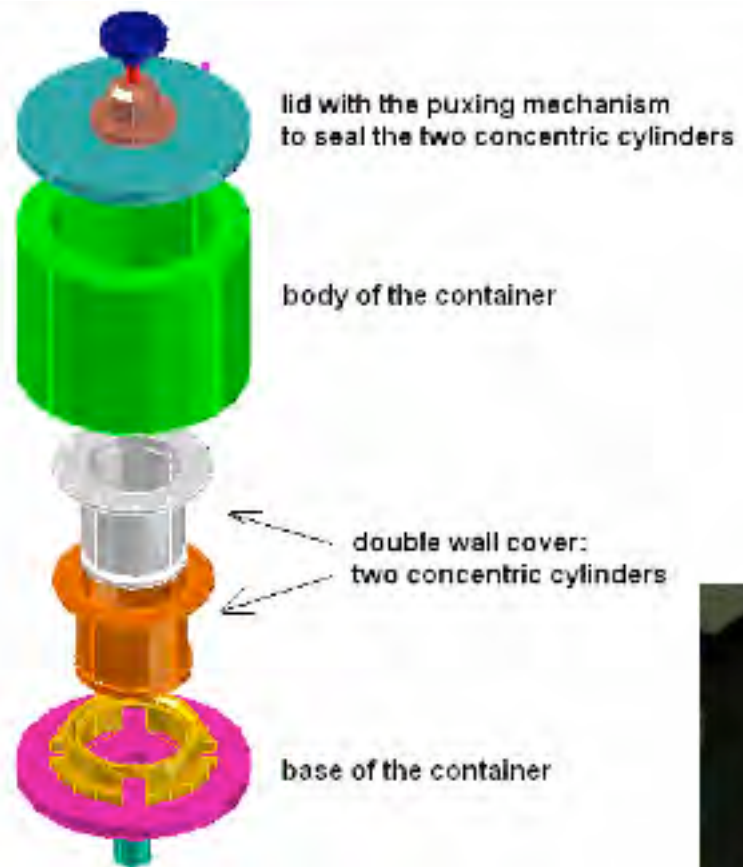
Improvements necessary on the mechanical part to reach 100% RH and ensure stability:

- double wall
- better isolation of water and upper part
- Change heating system of cover

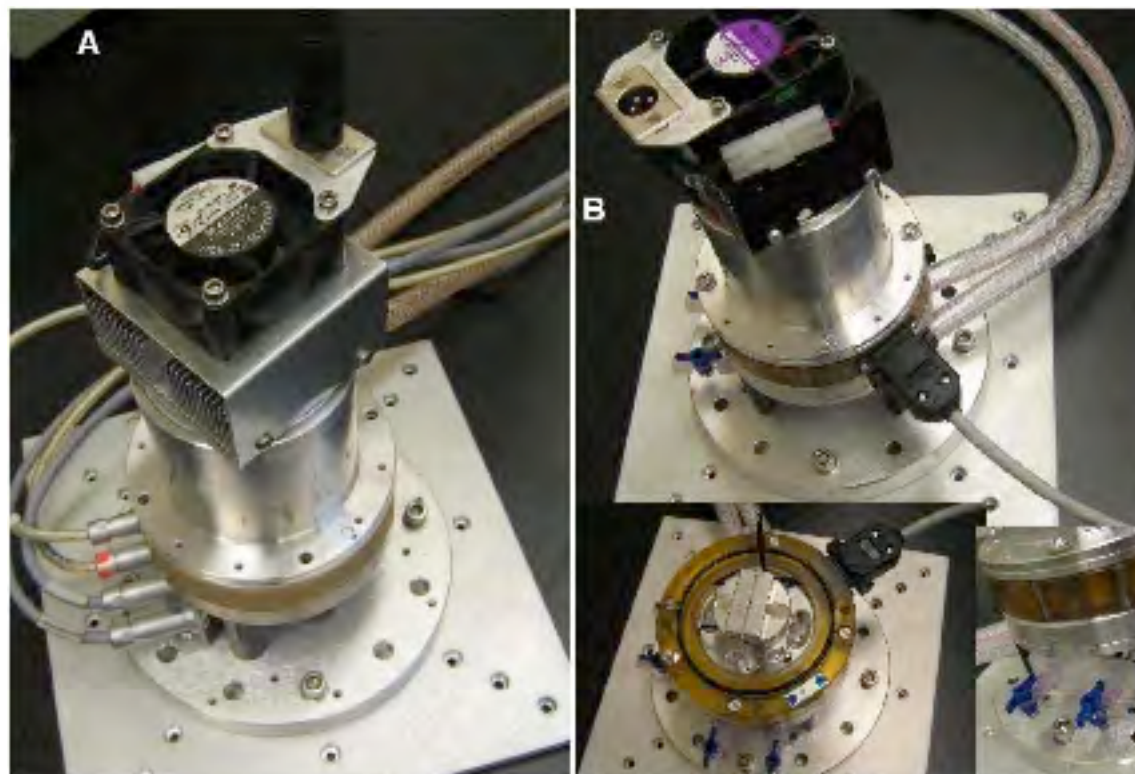
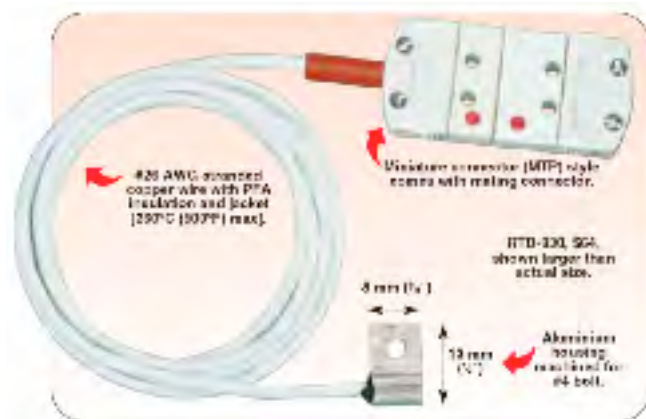
Setting of PIDs for various T and RH

$$r.h. = 100p(T_w)/p(T_s)$$

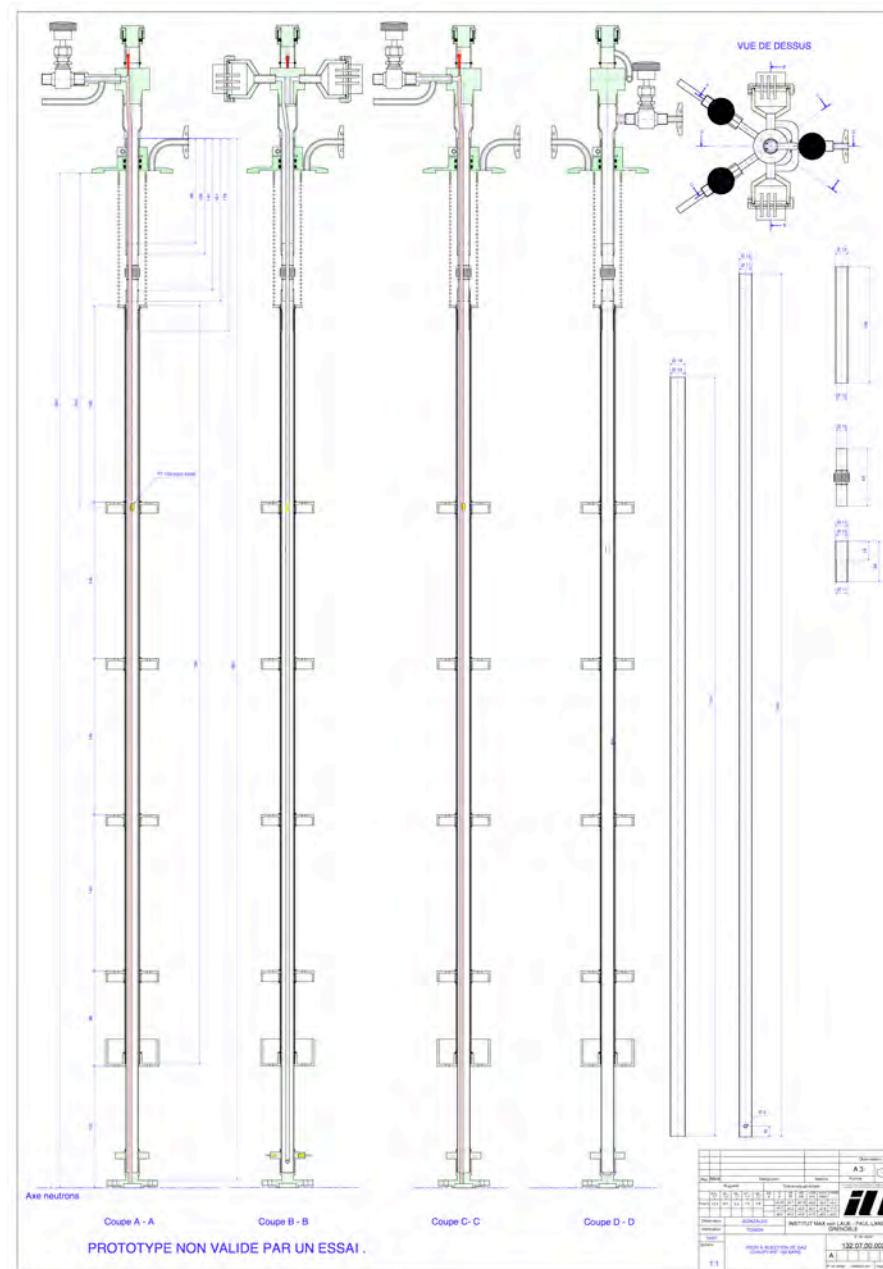
Electronics by Nadir Belkier
 Optimization by Leide Cavalcanti



Modification at University of Missouri (M. Rheinstädter and L. Cavalcanti)

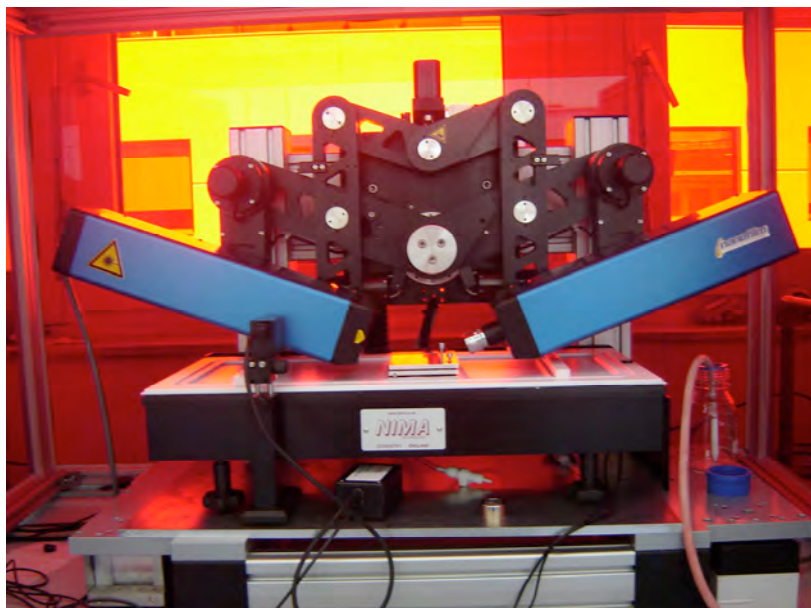


Developments for low T
 2 sticks 200bar
 Gas injection and
 humidity control
 Permanent flow or static
 measurements



Combined measurements:

FIGARO: Brewster Angle Microscopy



- Simultaneous measurements of in-plane features down to $2\mu\text{m}$

D22: Rheometry

- Shear response of complex fluids
- High torque measurements, small samples, volume and space accessibility
- T range -60°C 250°C (polymer composites and melts)
- Titanium and quartz bobs and cups

D11: Light Scattering

- Wide q-range explored in less time
- Control of sample status during experiment

What technical support?

- Room temperature expertise : stability of T controllers and optimization of sample holders, calibration of probes
- Mechanical work for dedicated sample environment to be shared between instruments (*instrument technician*)
- Help with off-the-shelf equipment : sample environment (rheometers, Langmuir troughs, stopped-flow, ...) and Soft Matter Lab (ellipsometer, Light Scattering, ...) - *mechanical work for dedicated sample environment (lab technician)*