The Institut Max von Laue –Paul Langevin

Welcome!

X-Ray and Neutron Science

3rd ESRF/ILL

International Student Summer Programme

28 August – 23 September 2016

W G Stirling
Director, ILL

The Institut Max von Laue –Paul Langevin

The European Neutron Source

Outline

- The EPN Science Campus
- Why neutrons?
- Neutron sources worldwide
- The Institut Max von Laue – Paul Langevin
- Science at ILL
- The future: Millennium and Endurance upgrade programmes

Grenoble

The Mountains

Grenoble : Wikipedia

- The city benefits from the highest concentration of strategic jobs in France after Paris, with 14% of the employments, 35,186 jobs, 45% of which specialized in design and research
- Grenoble is also the largest research center in France after Paris with 22,800 jobs (11,800 in public research, 7,500 in private research and 3,500 PhD students)
- In order to foster this technological cluster university institutions and research organizations united to create the GIANT (Grenoble Innovation for Advanced New Technologies) Innovation Campus with the aim at becoming one of the world's top campuses in research, higher education, and high tech

A little history: Grenoble has been transformed by science and technology

NEUTRONS FOR SCIENCE®

1956 CEA (CENG)

1962 CNRS

1967 ILL

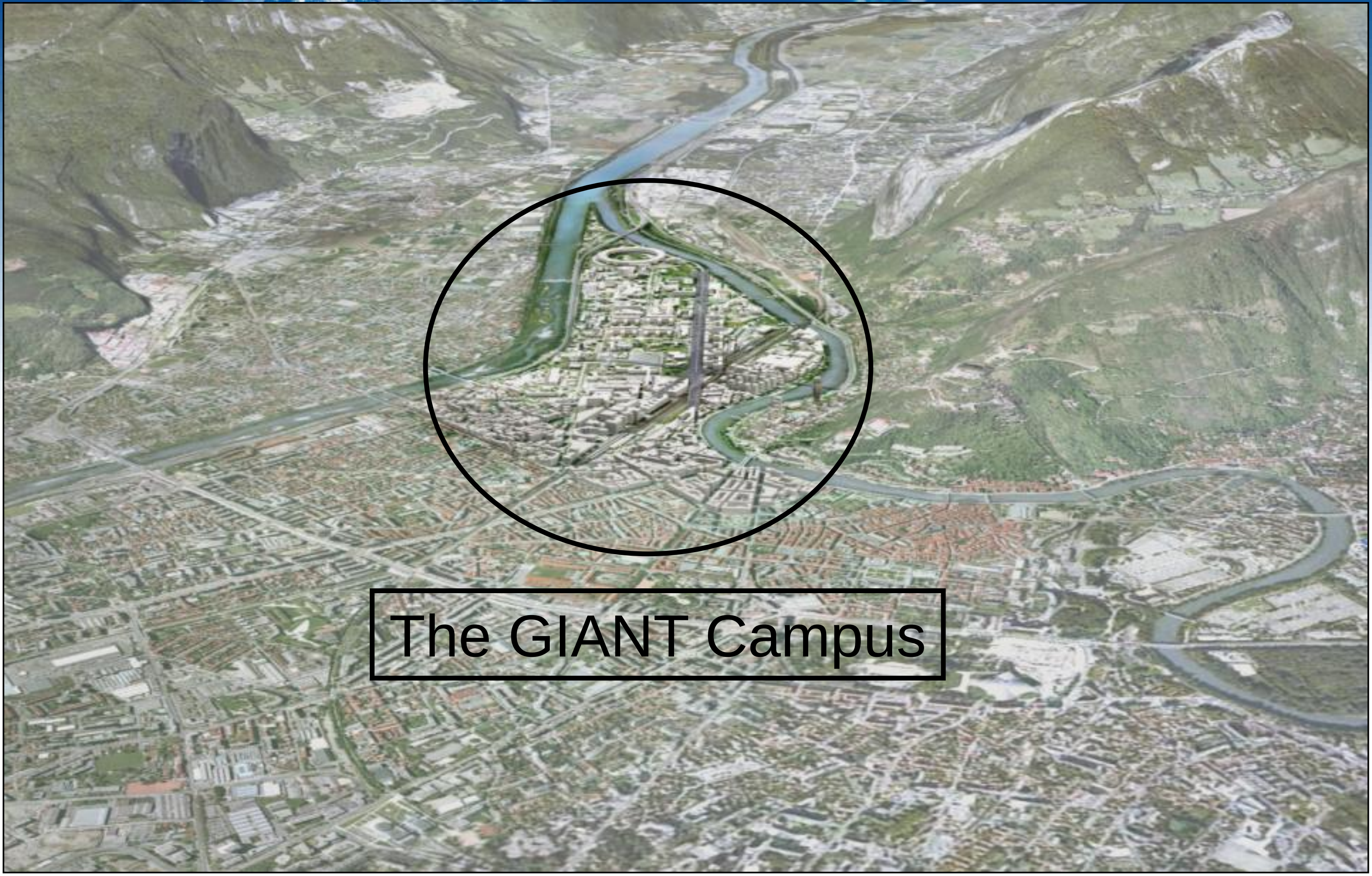
and *l'Université Joseph Fourier (now UGA), Grenoble Institut Polytechnique, Grenoble Ecole de Management* …

1988 ESRF
European Synchrotron
Radiation Facility

2005 MINATEC
Micro and Nano-electronics

2009 GIANT
Innovation Partnership

Grenoble: the GIANT campus



GIANT: Grenoble Innovation for Advanced New Technologies

Grenoble: the GIANT campus

The EPN Science Campus

EPN Science Campus

Institut Laue-Langevin (ILL) operates the most intense (reactor) neutron source in the world, feeding a suite of 40 high-performance instruments

European Synchrotron Radiation Facility (ESRF) is a world-leading synchrotron radiation source hosting 50 cutting-edge experimental stations

The Institut de Biologie Structurale (IBS) is a research centre in structural biology. The IBS possesses cutting edge facilities and is a partnership between CEA, CNRS and UJF

EMBL Grenoble is an outstation of the EMBL organisation (HQ in Heidelberg), specialising in research in structural biology (in very close proximity to the ILL and the ESRF)

Why neutrons?

Thermal neutrons

- Uncharged particles (waves):

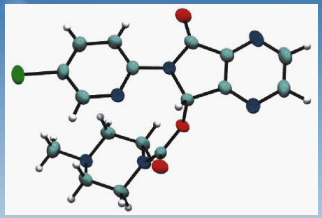
  produced by fission (reactors) or

  spallation (accelerators, pulsed sources)
- $E \sim k_B T$ (so typically meV)
- Wide range of energy/wavelength:

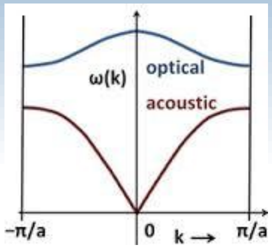
  from μeV (~ 30 nm = 300Å) to

  eV (~ 0.03 nm = 0.3Å)
- Spin ½ so can be spin-polarised
- Penetrate materials

Why are neutrons useful for studying materials?

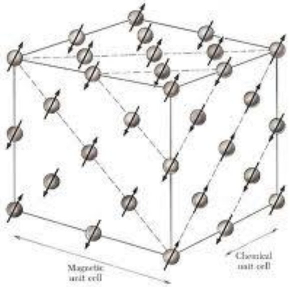


- Wavelength (Å) ~ interatomic spacings
  Diffraction → structures at atomic level



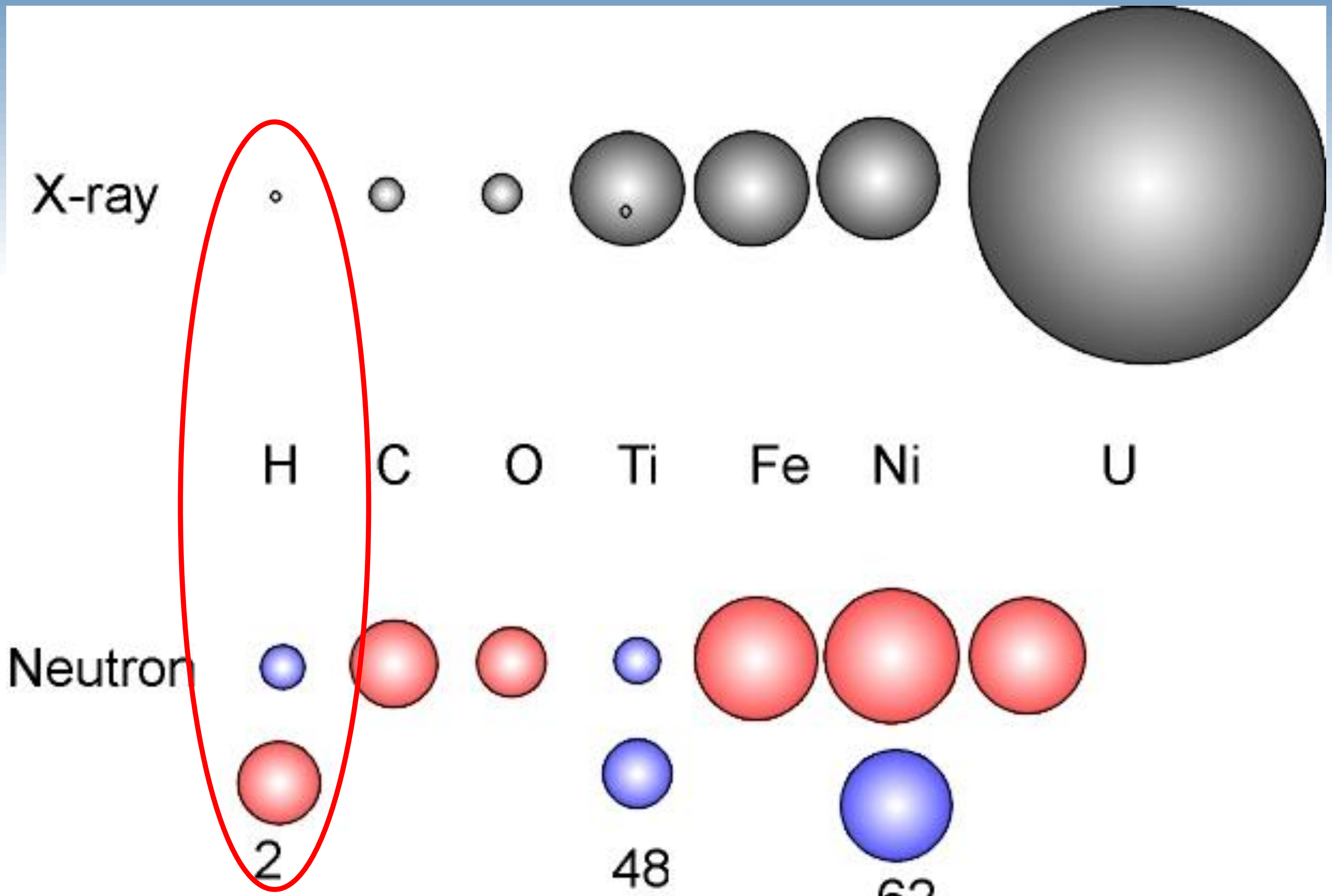
- Energy (meV) ~ phonon/spin-wave energies
  Inelastic scattering → dynamics

- Scattering power does not vary simply with Z
  Isotopes → different scattering (contrast e.g. H and D)
- Neutron spin → magnetic information (structures and dynamics)

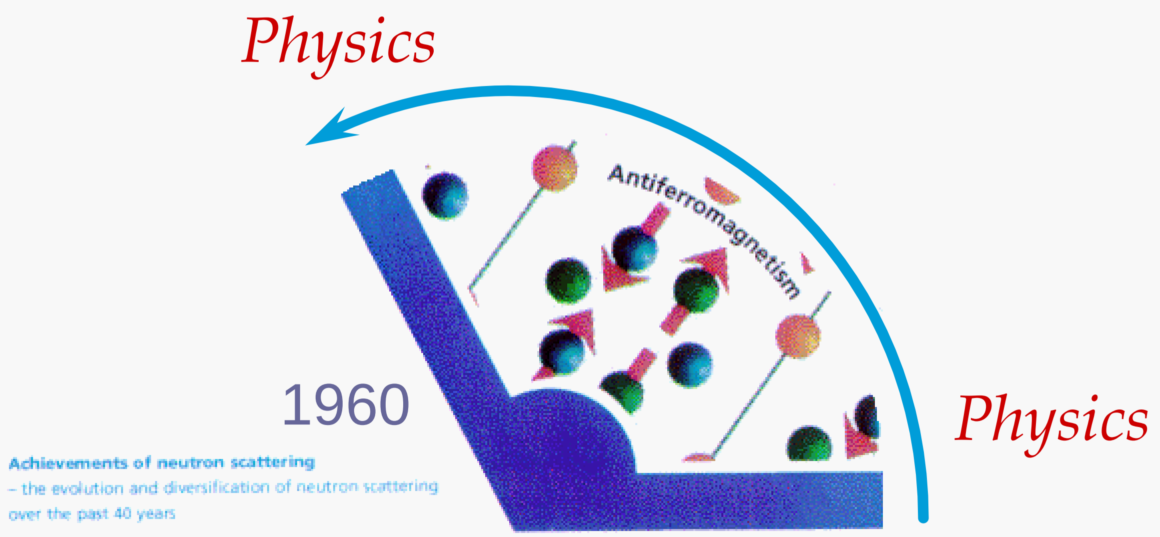


Scattering power does not vary simply with Z

Scattering is different for different isotopes of same element (H and D)

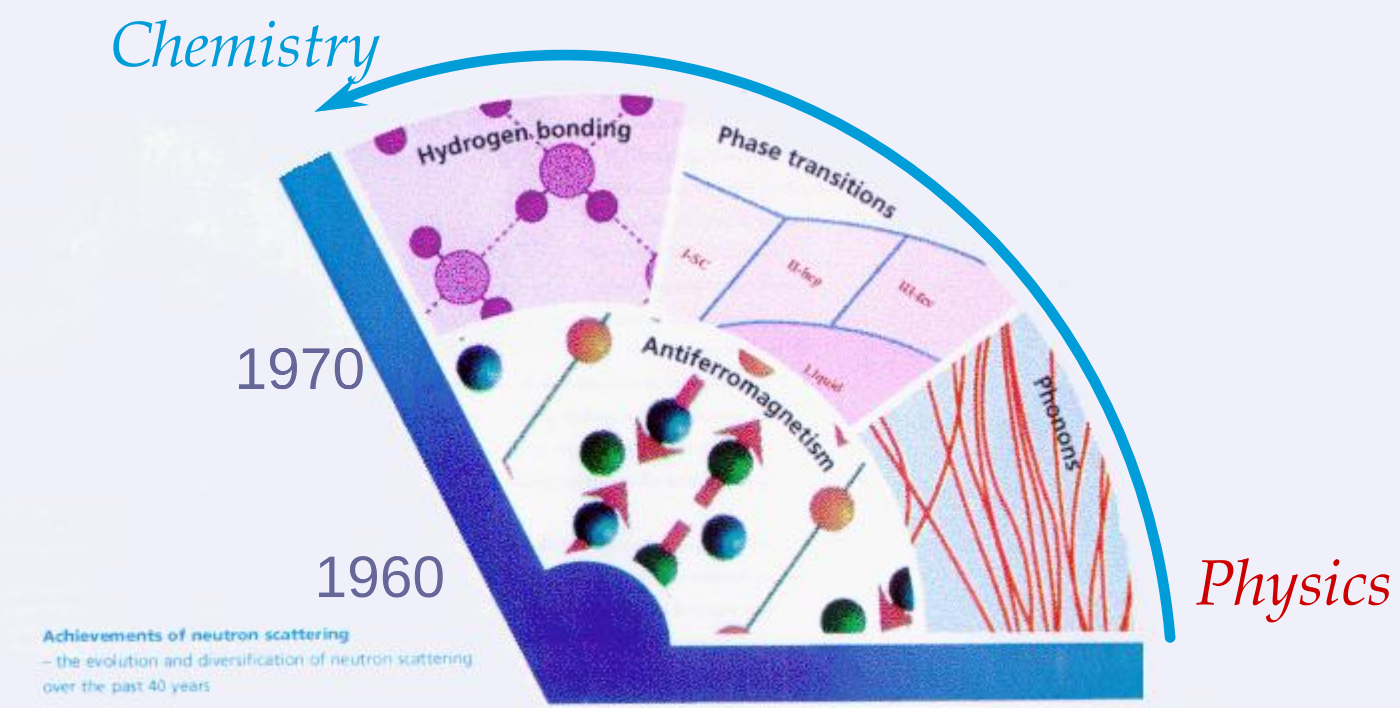


Science with neutrons



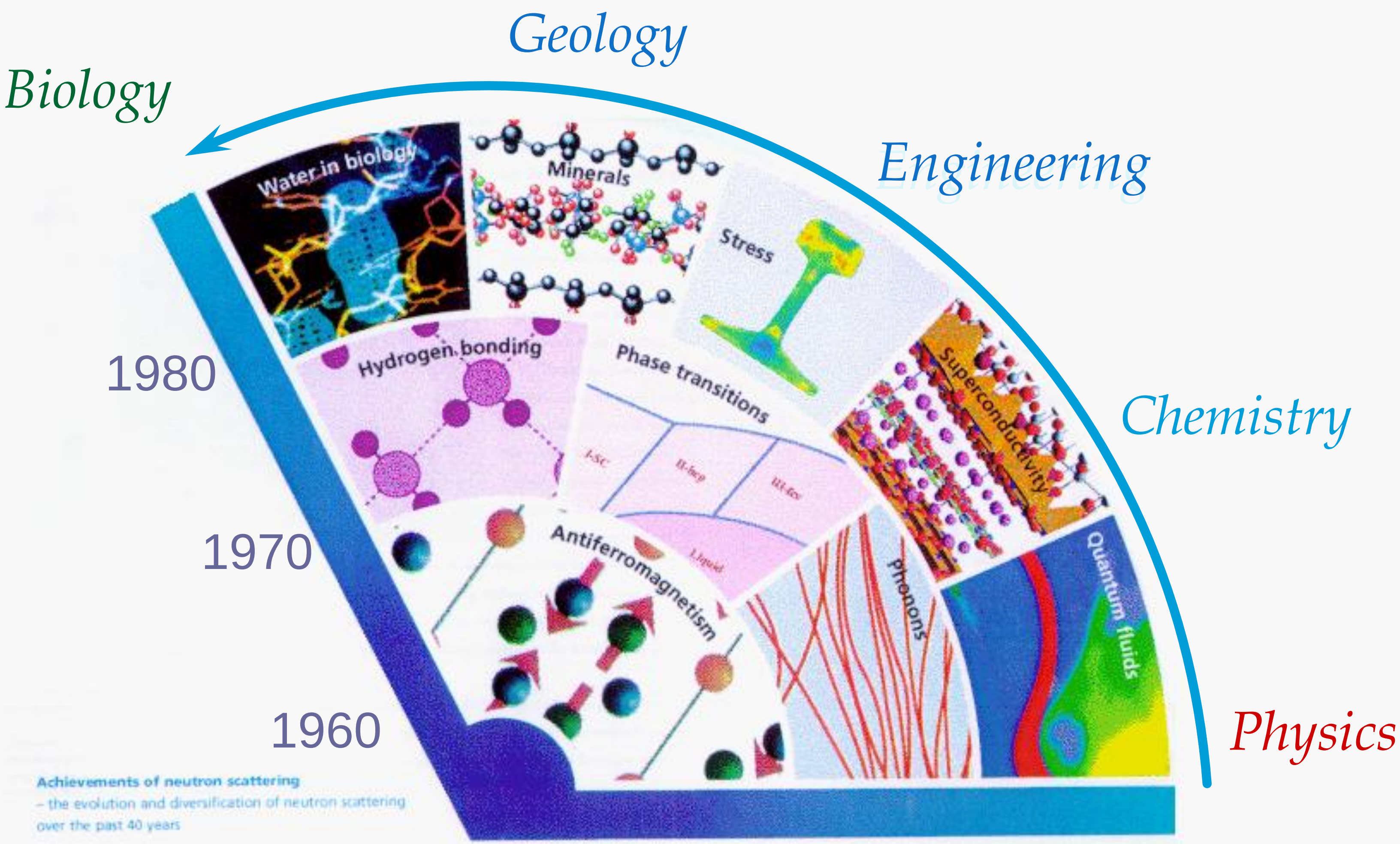
Courtesy of ISIS

Science with neutrons



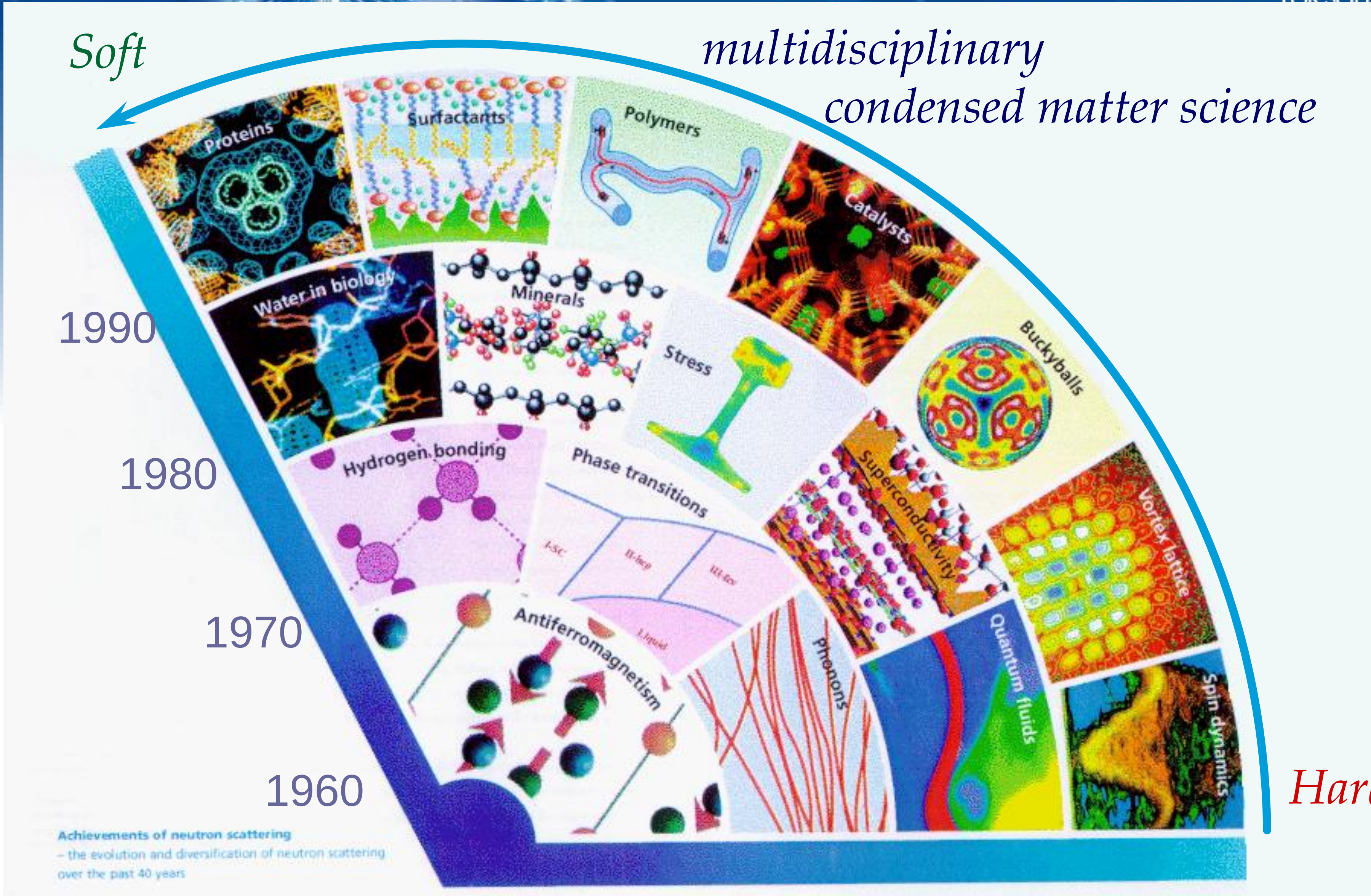
Courtesy of ISIS

Science with neutrons



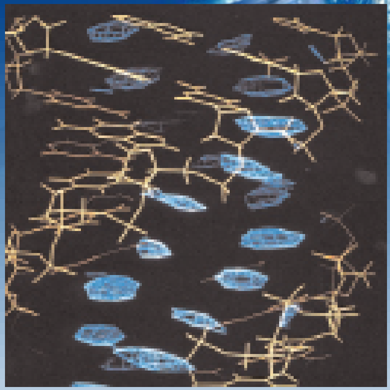
Courtesy of ISIS

Science with neutrons

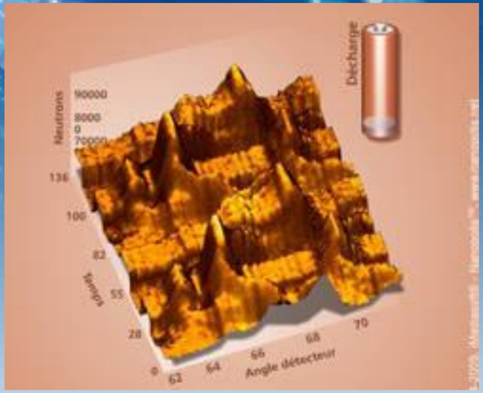


Courtesy of ISIS

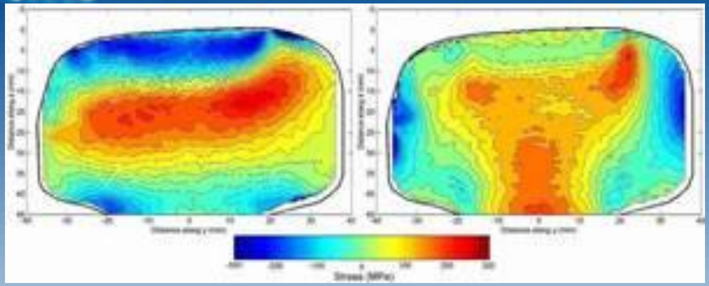
Science and technology with neutrons



Biology/life sciences



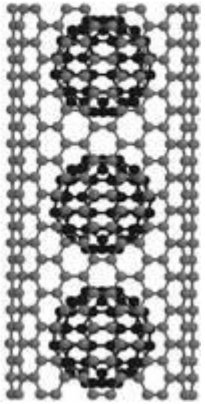
Chemistry



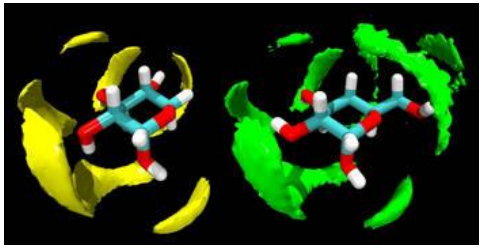
Engineering

Growth fields: soft condensed matter (kinetics, colloids, chemical processing), materials/engineering, imaging, cultural/heritage …

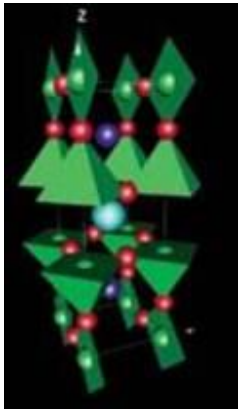
Materials



Liquids

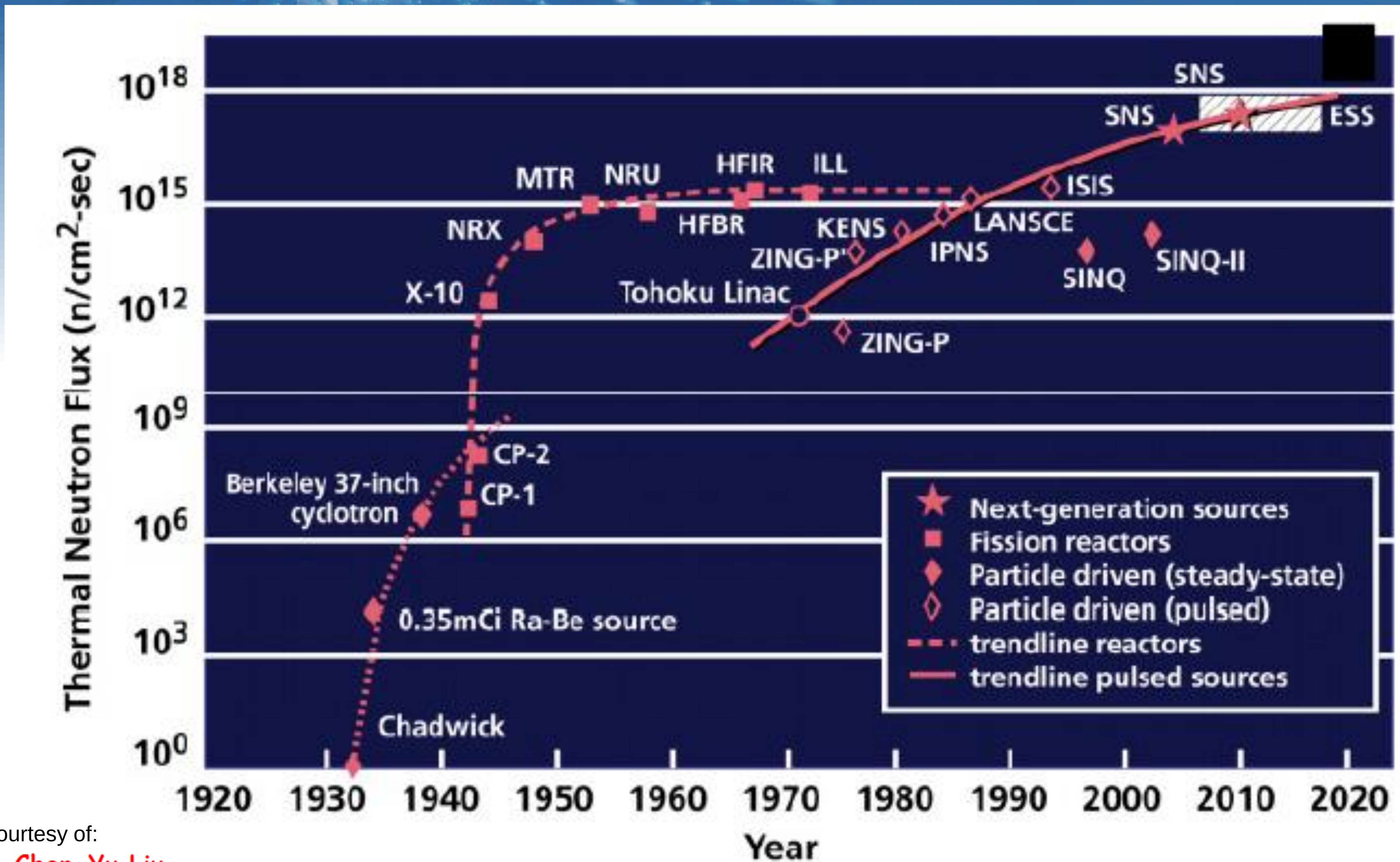


Magnetism



Nuclear/fundamental physics

History of Neutron Sources



Updated from *Neutron Scattering*, K. Skold and D. L. Price: eds., Academic Press, 1986)

Courtesy of:
Chen-Yu Liu
CL21@indiana.edu
Indiana University

Neutron Sources Worldwide

➢ Europe (~18)	France, Germany, UK, Russia…
➢ North America (~10)	USA, Canada
➢ Oceania (1)	Australia
➢ Asia (~12)	Japan, China, India, Korea, Malaysia, Indonesia
➢ South America (1)	Argentina (Brazil)

Neutron Sources Worldwide

ILL

ISIS

SNS

HFIR

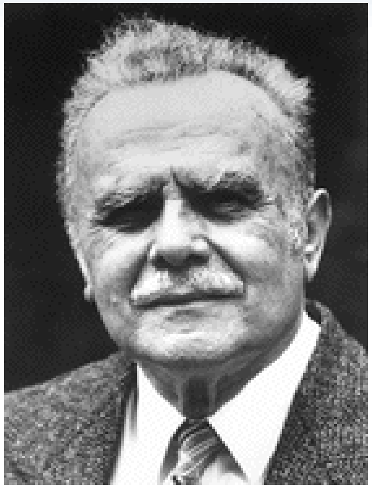
The big facilities:

- ILL, Grenoble; ISIS, Oxford, UK; SNS and HFIR, ORNL, USA
- NIST, Washington, USA; Orphée, LLB, Saclay, France; FRM-II, Garching, Germany; OPAL, Lucas Heights, Australia; J-PARC, Tokai, Japan
- Future: ESS, Lund (first neutrons: 2019-2020)

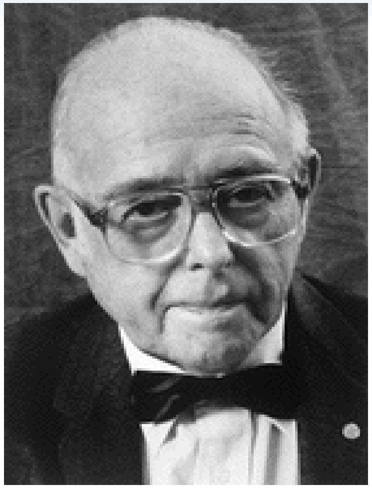
Nobel Prize in Physics, 1994

A unique probe of ‘where atoms are and what atoms do’ -

to paraphrase the citation for the Nobel Prize in Physics awarded to Brockhouse and Shull in 1994



Bert Brockhouse
dynamics



Cliff Shull
structure

The Institut
Max von Laue – Paul Langevin

ILL: a little history …

- Founded in 1967 (France, Germany)
- UK joined in 1974 (3 Associates)
- 9 Scientific Member countries

19 January 1967: signature of agreement between France and Germany

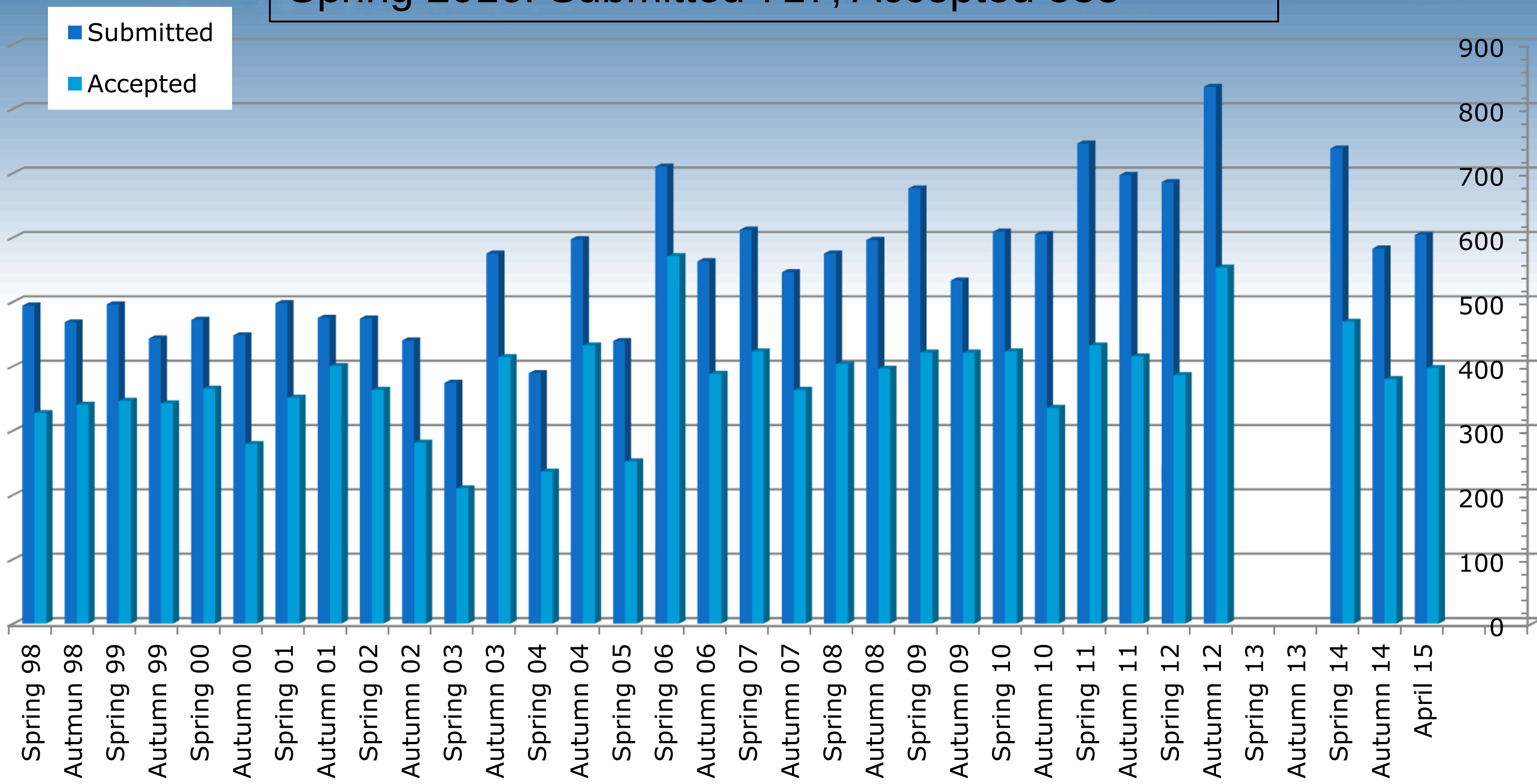
Louis Néel and Heinz Maier-Leibnitz

ILL: a little history …

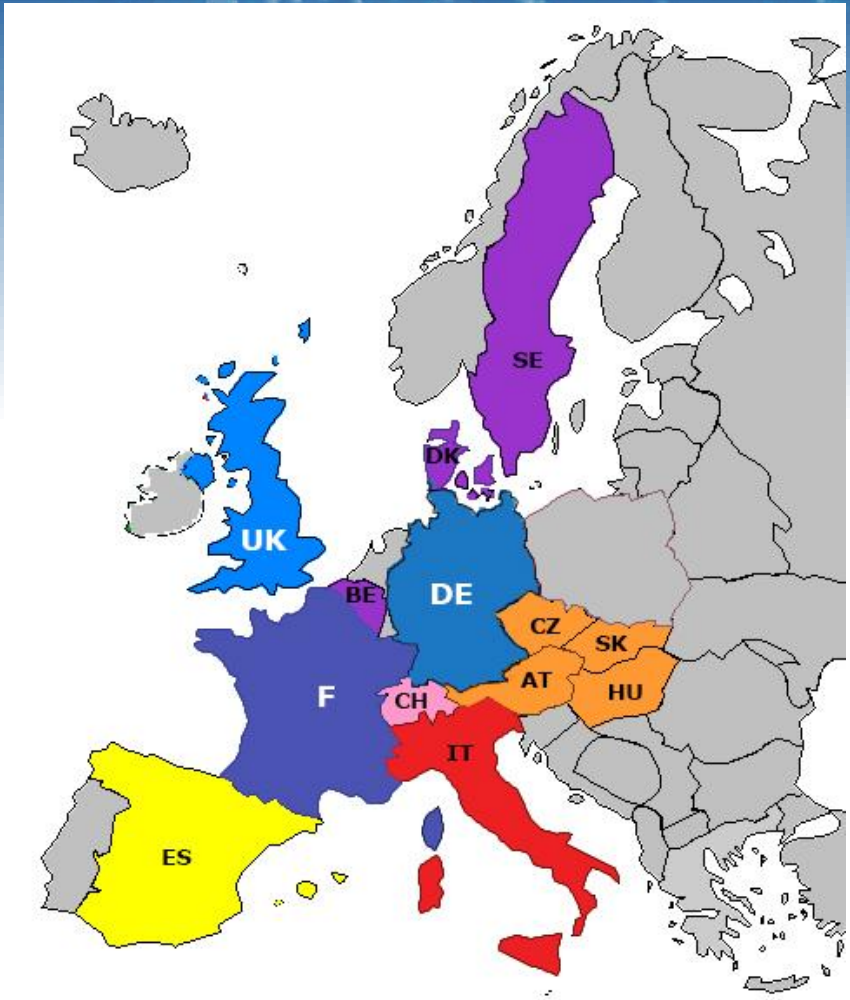
- ILL was the first major international scientific **User** facility (? CERN)
- University and research centre scientists (Europe and world-wide) write research proposals to use ILL's neutron scattering and fundamental/nuclear physics instruments
- Proposals considered twice each year by **external** expert panels (peer review), after internal technical review (feasible?)
- Accepted proposals get beamtime (free for Member countries!)
- Travel and subsistence costs for experimental team covered by ILL (Member countries)
- Proposers expected to **publish** results in scientific literature
- Industry can buy beamtime for proprietary research (not expected to publish)

Submitted and accepted proposals

Spring 2016: Submitted 717; Accepted 538



ILL member countries



Associates (budget)

Germany:	25 %
UK:	25 %
France:	25 %

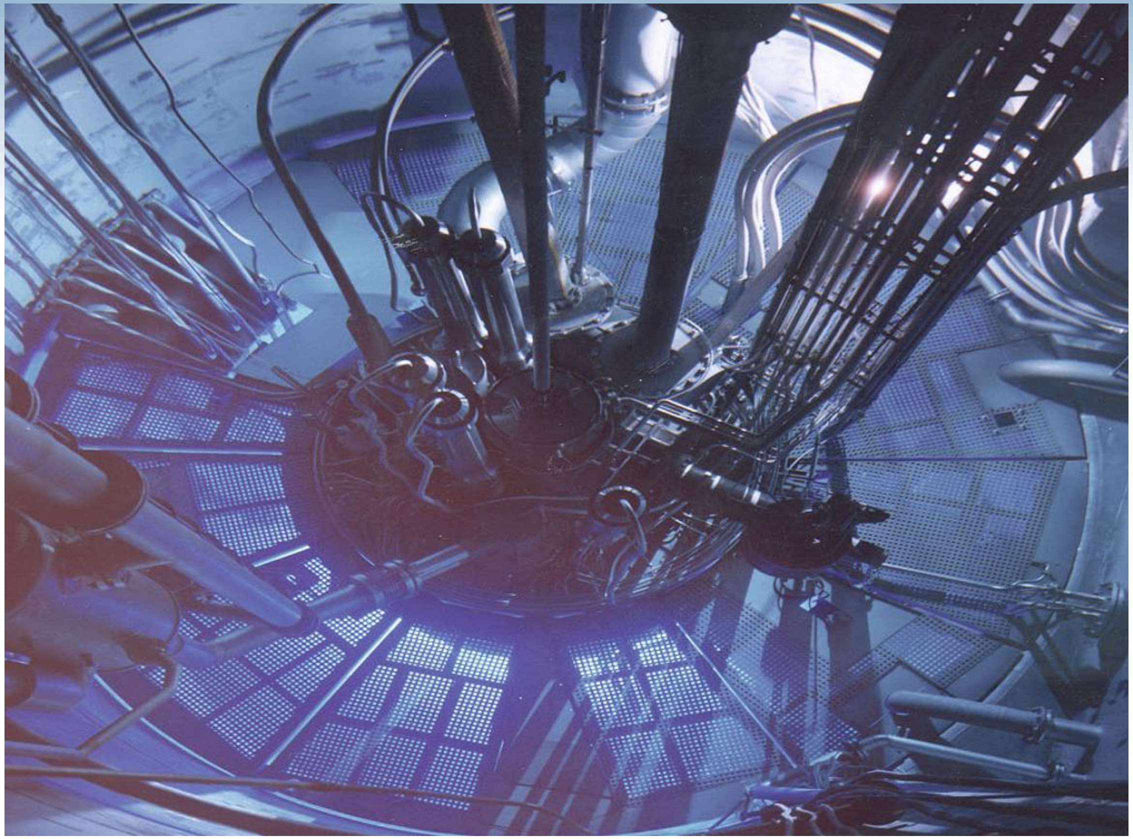
Scientific Members (25%)
Spain, Italy, Switzerland ,
Denmark, Austria,
Czech Republic, Slovakia,
Belgium, Sweden

(Poland, Hungary)

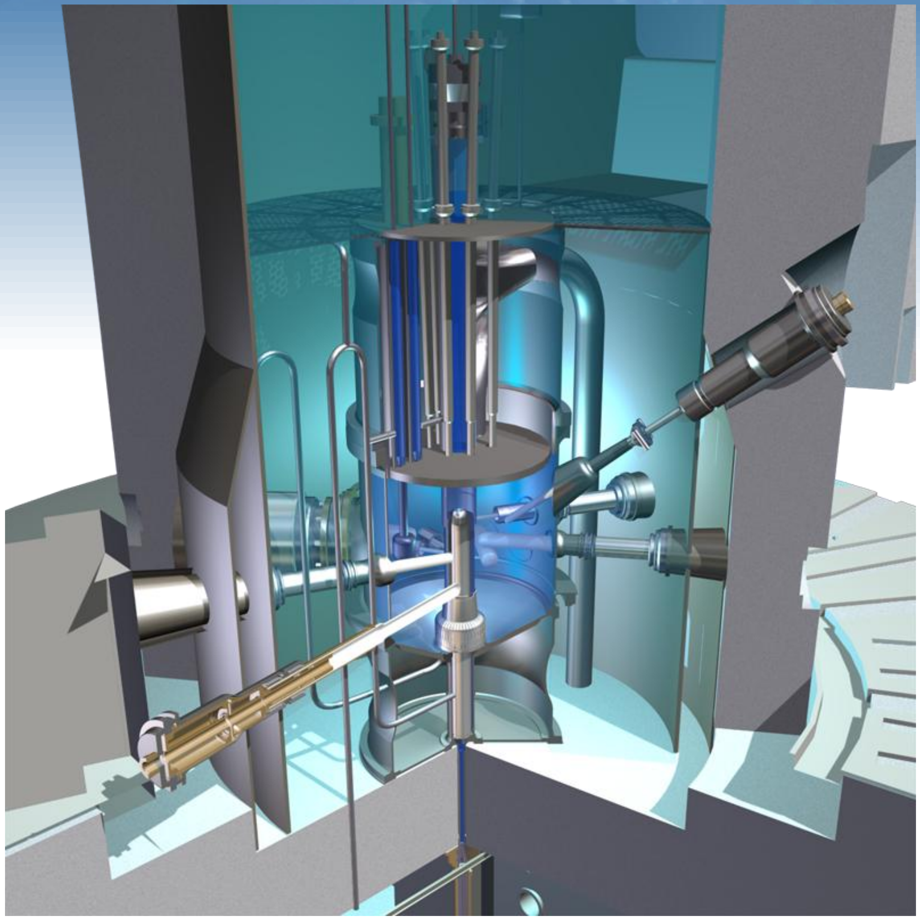
(India: until 2015)

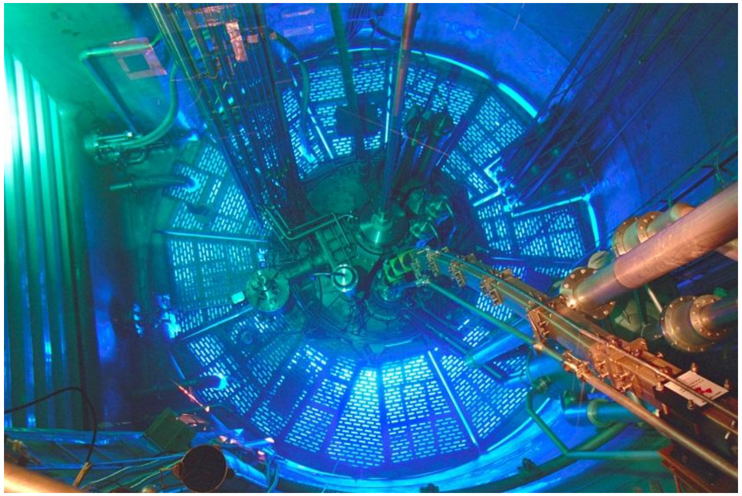
ILL: the world leader. Why?

Highest flux (power ~ 58 MW) – plus hot and cold sources



The ILL High Flux Reactor



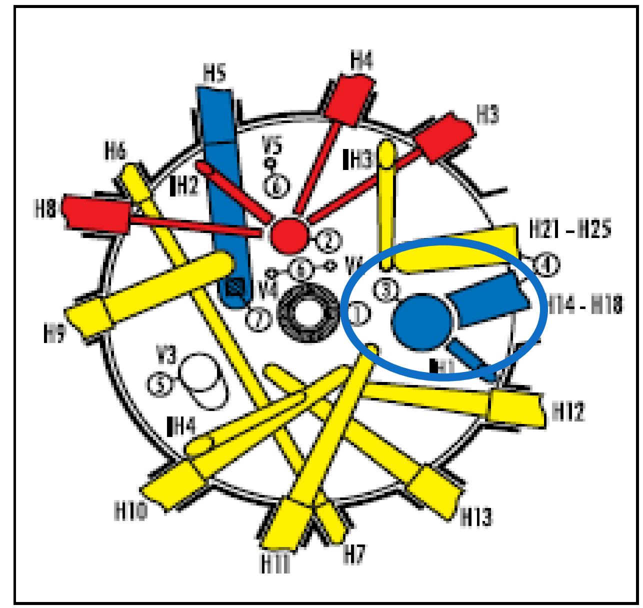


58 MW reactor operating ~200 days/year; 4 Cycles of ~50 days /year

Cold and Hot Sources (ILL)

Vertical Cold Source

- Al sphere 38 cm diameter
- 20 litres of boiling D_2 at 25K
- Enhances intensity for λ > 3Å
- **Shifts neutron spectrum to longer λ, lower E**
- And also Horizontal Cold Source (in beamtube)

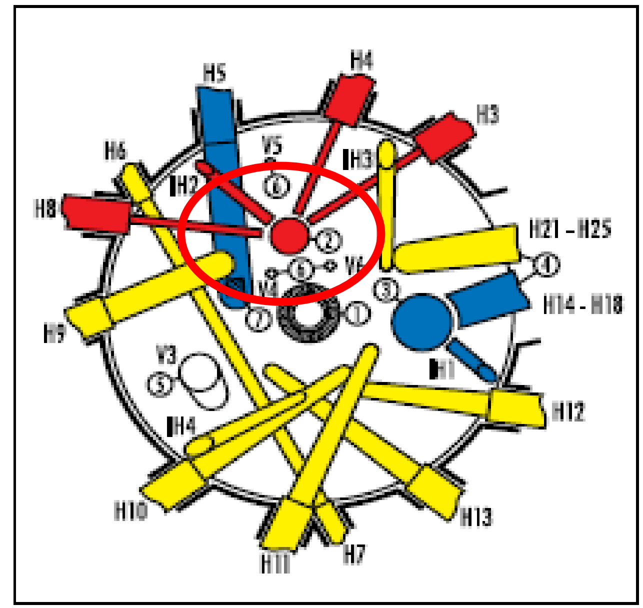


Beam-tube arrangement at the ILL-HFR

Cold and Hot Sources (ILL)

Hot Source

- Block of graphite, 10 litres
- Heated (nuclear) to 2400K
- Enhances intensity for $\lambda <$ 0.8Å
- **Shifts neutron spectrum to shorter λ, higher E**



Beam-tube arrangement at the ILL-HFR

ILL: the world leader. Why?

28 public instruments + 9 CRGs

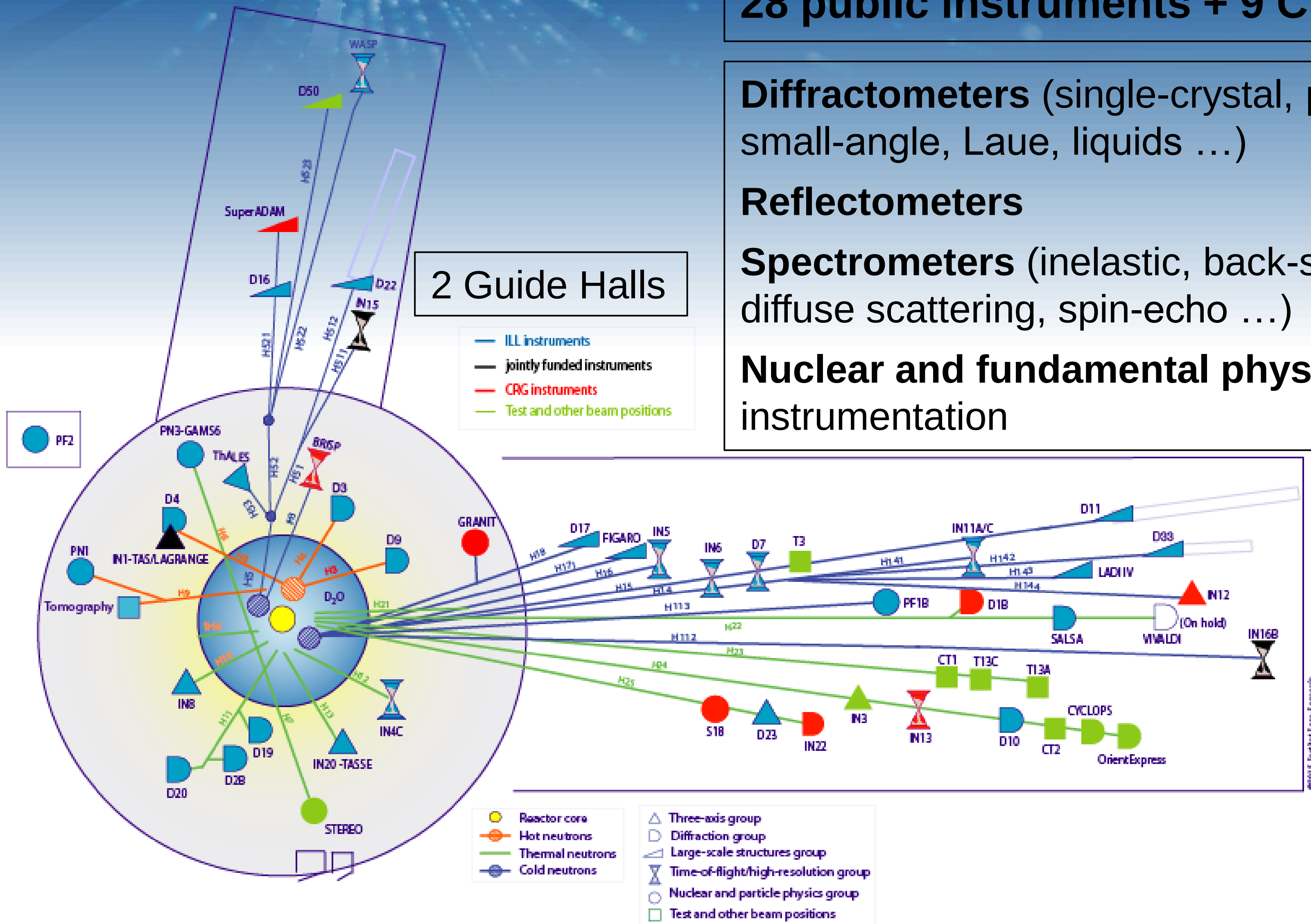
Diffractometers (single-crystal, powder, small-angle, Laue, liquids …)

Reflectometers

Spectrometers (inelastic, back-scattering, diffuse scattering, spin-echo …)

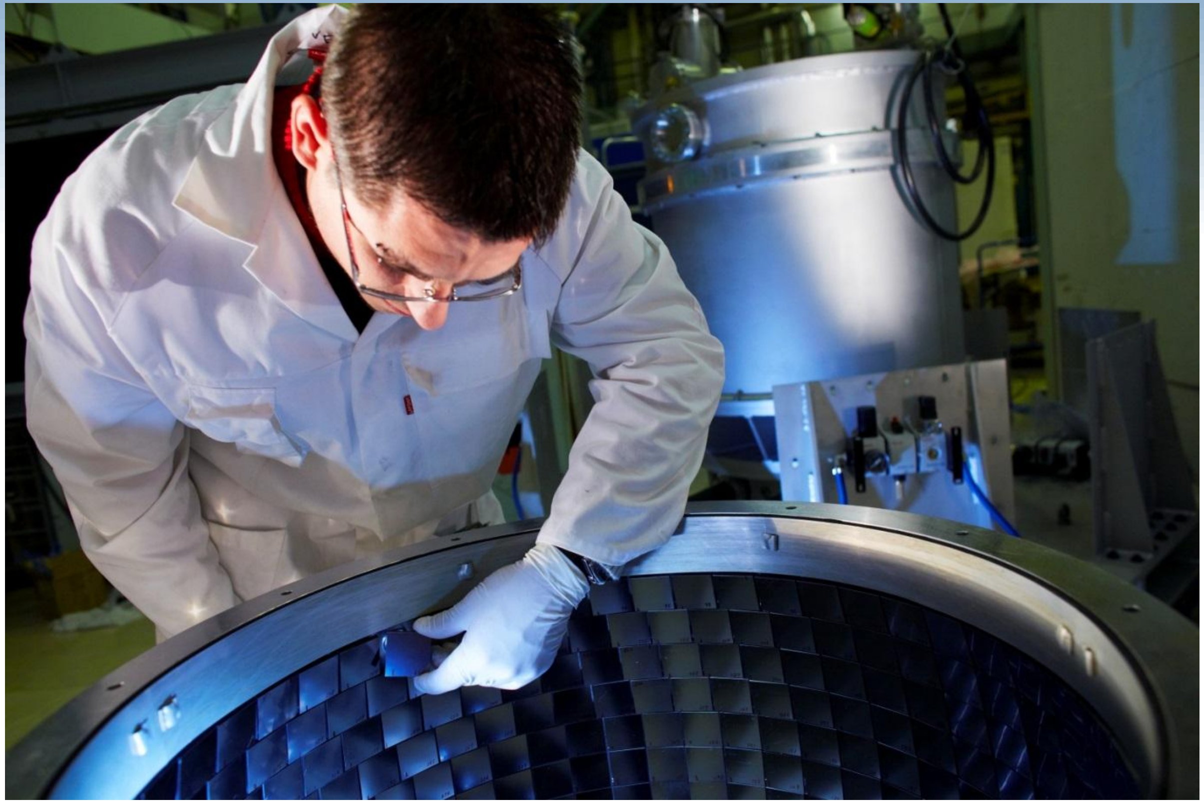
Nuclear and fundamental physics instrumentation

2 Guide Halls



ILL: the world leader. Why?

Highly qualified / highly trained staff



~ 470 staff

~ 80 scientists

~ 85 engineers

~ 35 thesis students

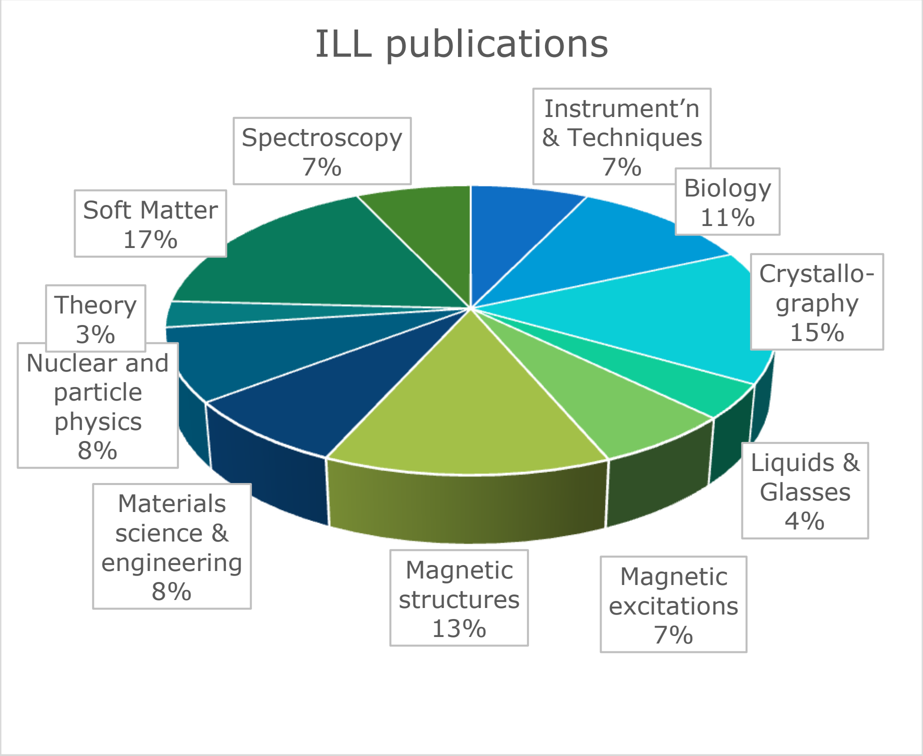
~ 200 technicians

~ 70 administration/ support

Science at ILL

Science at the ILL

- 1200 proposals:
  9000 instrument days
- 1500 - 2000 users
- 850 experiments/year;
  5000 – 6000 instrument days
- 40 countries
- 28 instruments + 9 CRGs
- 550 - 600 publications/year



Biophysical studies of Moringa protein for improved water purification

NEUTRONS FOR SCIENCE®

- Moringa protein used in traditonal water purification for hundreds of years
- Neutron studies by scientists from Namibia, Botswana, Uppsala, Keele, ILL
- Neutron research optimising Moringa use → cheap, effective purification in Africa

Moringa oleifera tree (Miracle tree)

Seeds

Traditional preparation

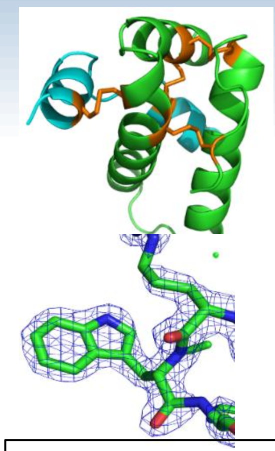
Water before and after Moringa treatment

Village scale use of Moringa protein

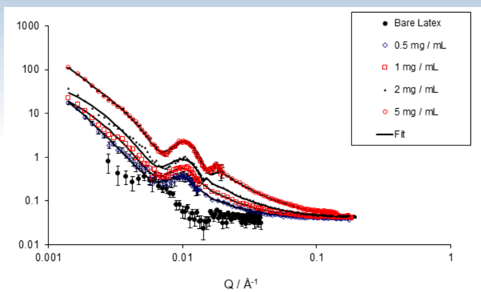
Biophysical studies of Moringa protein for improved water purification

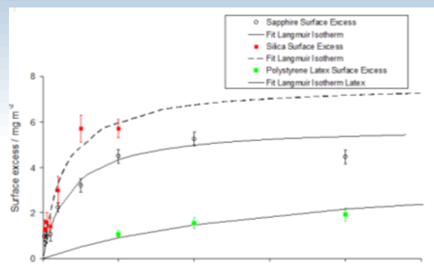
NEUTRONS FOR SCIENCE®

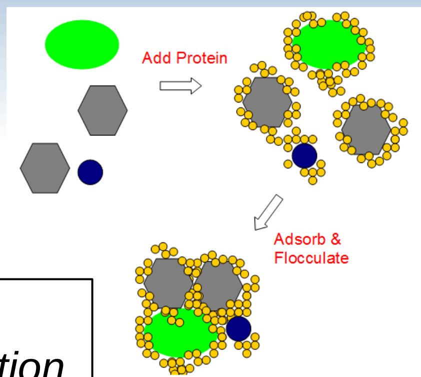
- Neutrons and Partnership for Structural Biology (PSB) platforms:
  - understanding structure and properties
  - rational optimisation
- Wider use evaluated by Namibian Government and Botswanan agencies

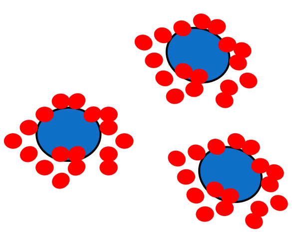


Molecular level : X-ray crystal structure of protein

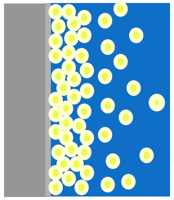








Multilayer of protein on different materials



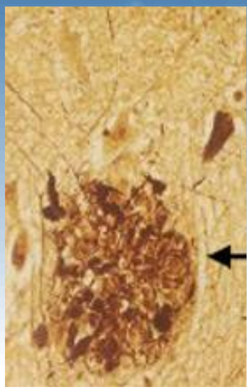
Optimise concentration

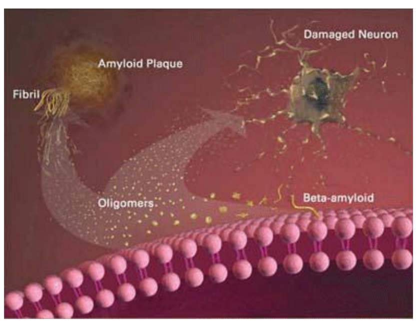
Model for action of protein

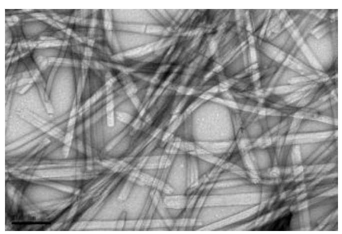
SANS — *Deuteration* — *Neutron reflectivity*

P S B

Structural studies of amyloid fibres





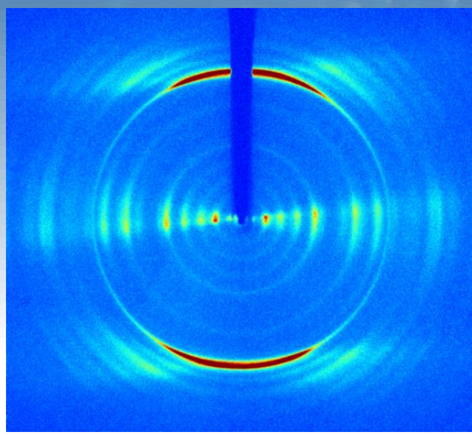


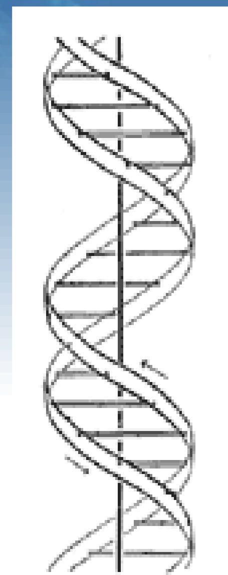
Amyloid plaques

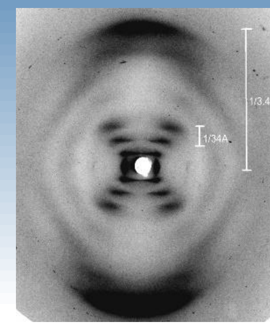
Amyloid fibrils by EM

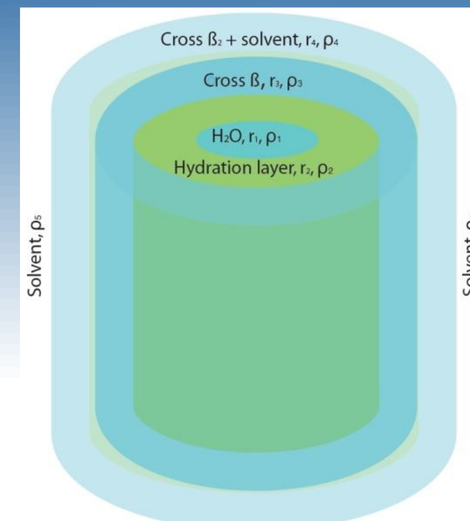
- Amyloids are insoluble fibrous deposits that arise from the incorrect folding of proteins
- Amyloidosis is associated with numerous neurodegenerative conditions including Alzheimer’s disease, Parkinson’s disease, Huntington’s disease.
- Neutron studies of these fibres are being used alongside synchrotron and FEL X-ray methods to probe the structure and assembly of these fibrillar structures.

Structural studies of amyloid fibres









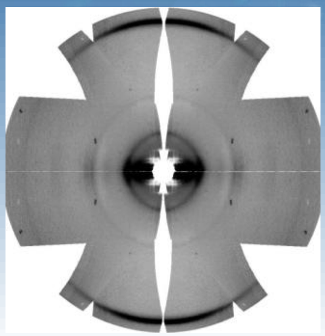
Fibre diffraction

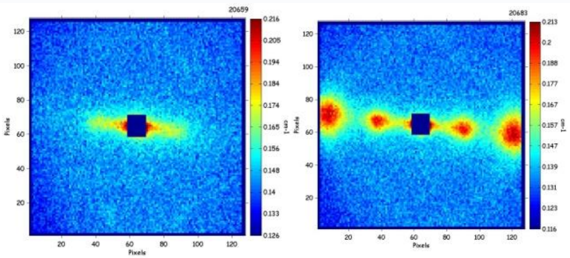
Fibre diffraction was the technique used to determine the structure the the DNA double helix

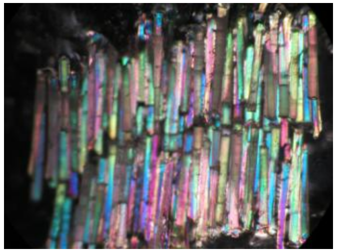
Models for structure and hydration

Levels of organisation/analysis for amyloid: from cellular to atomic

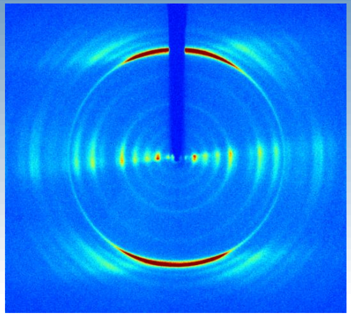
Structural studies of amyloid fibres

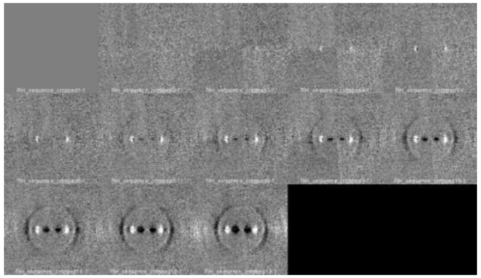




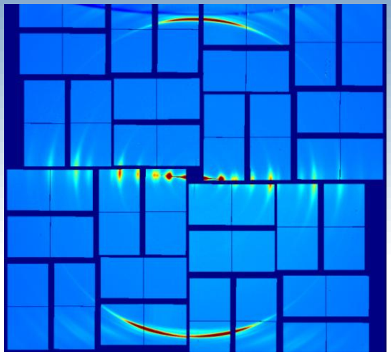


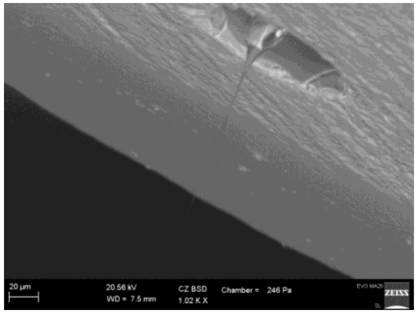
Neutrons (ILL): amyloid hydration





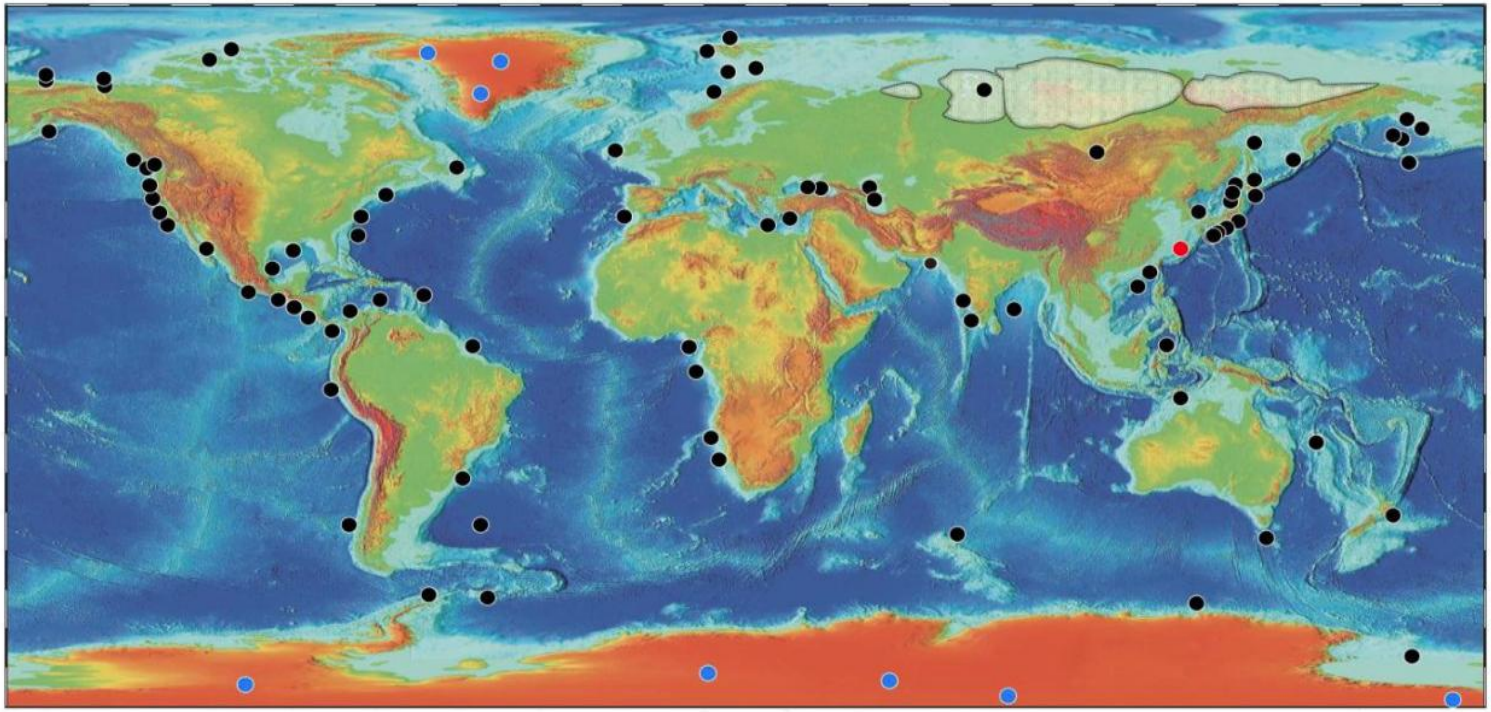
Synchrotron X-rays (ESRF): hydration driven structural transitions



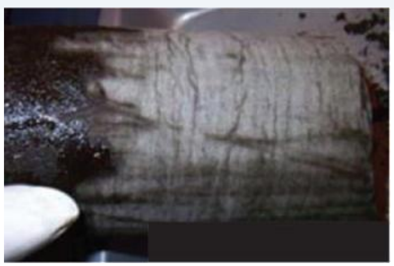


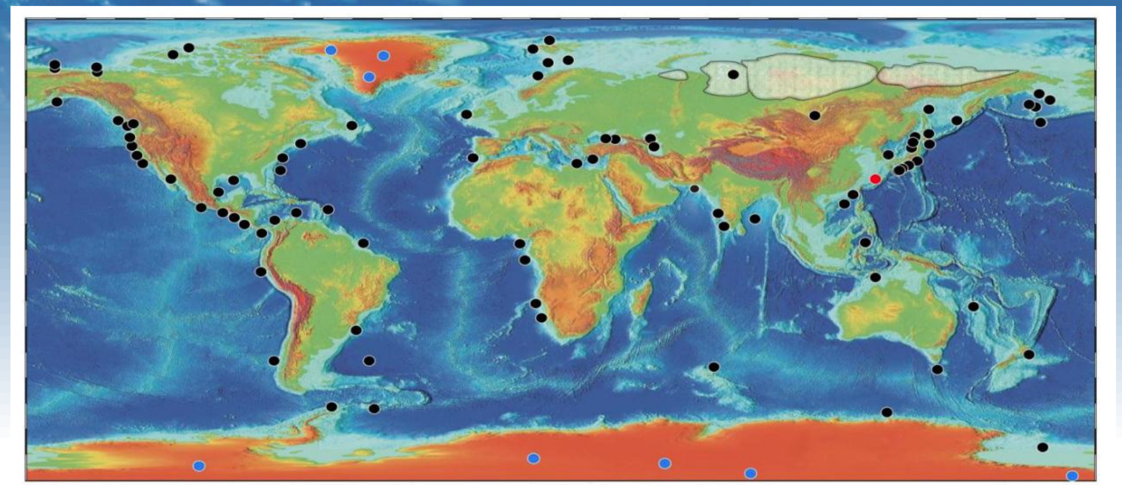
FEL X-rays (Stanford): single particle X-ray diffraction (collaboration with CFEL)

Formation of ice XVI
Powder Diffraction (neutrons)



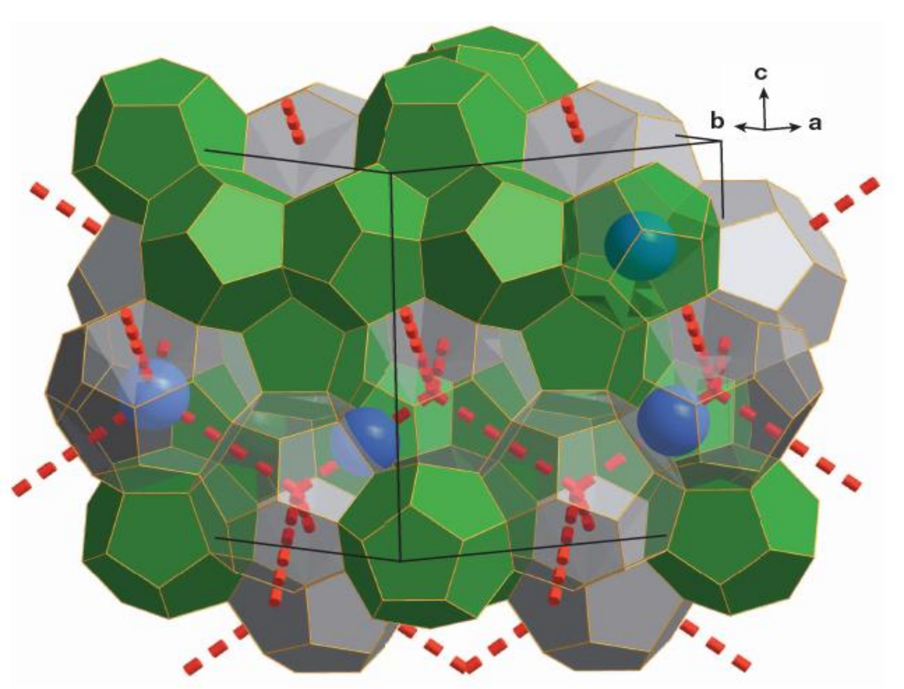
Formation of ice XVI





- Gas hydrates – ice-like solids, guest molecules inside cages within crystalline framework (clathrates) of H-bonded water molecules
- Deep ocean floor/permafrost: fossil fuel reserve (methane) and climate hazard
- **Empty** clathrate very important – thought to be experimentally inaccessible since guest molecules stabilise host framework

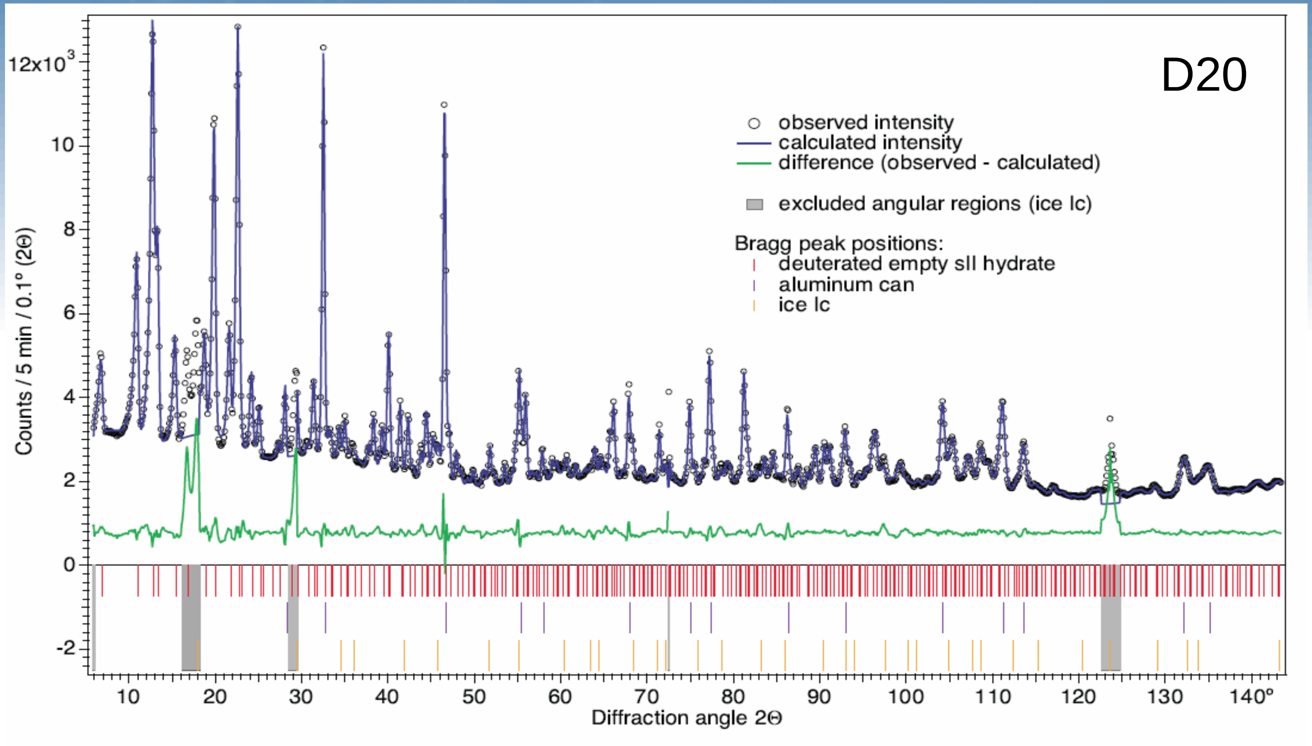
Formation of ice XVI



- The 17th ice phase discovered by emptying a type sII Ne clathrate hydrate
- Decades of speculation about existence of such a phase
- Falenty, Hansen and Kuhs, Nature 516, 231, 2014 (U Göttingen, ILL)

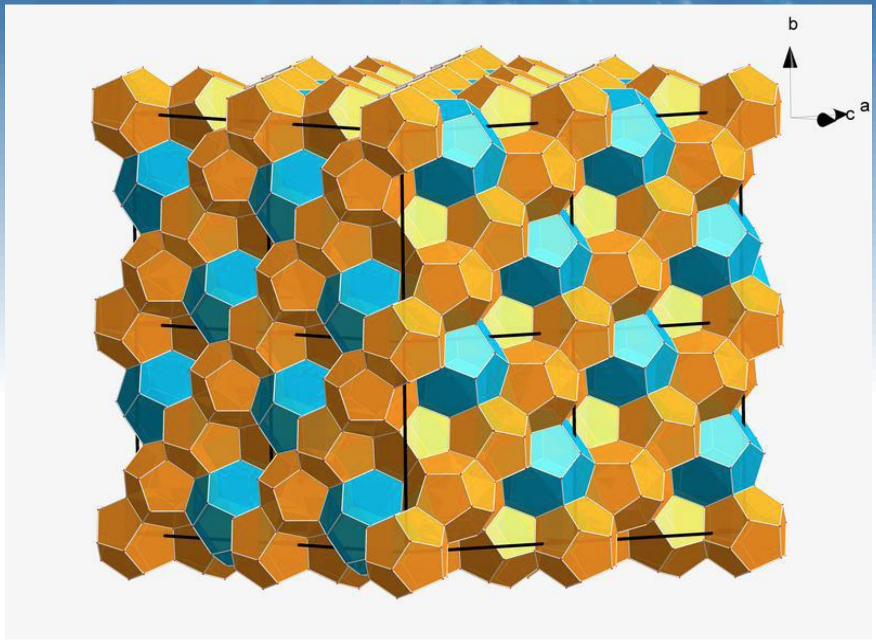
Ne atoms (blue) move out between large cages (grey) through 6-membered rings of water molecules (red dashed lines)

Formation of ice XVI

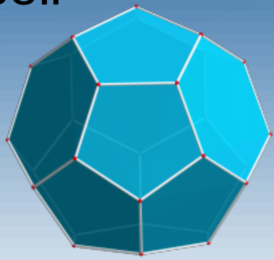


After 5 days of pumping at 142 K Ice XVI is born.

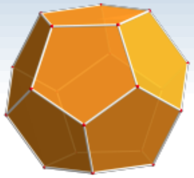
Formation of ice XVI



- 136 molecules per unit cell



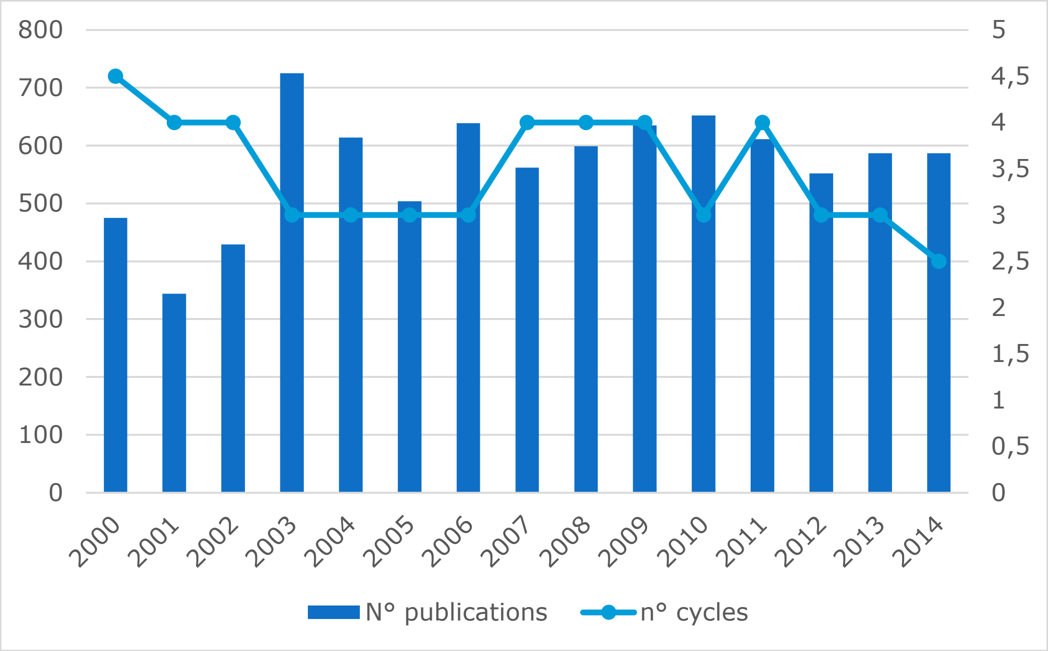
- 8 large cages connect via hexagonal faces



  16 small cages fill the remaining volume

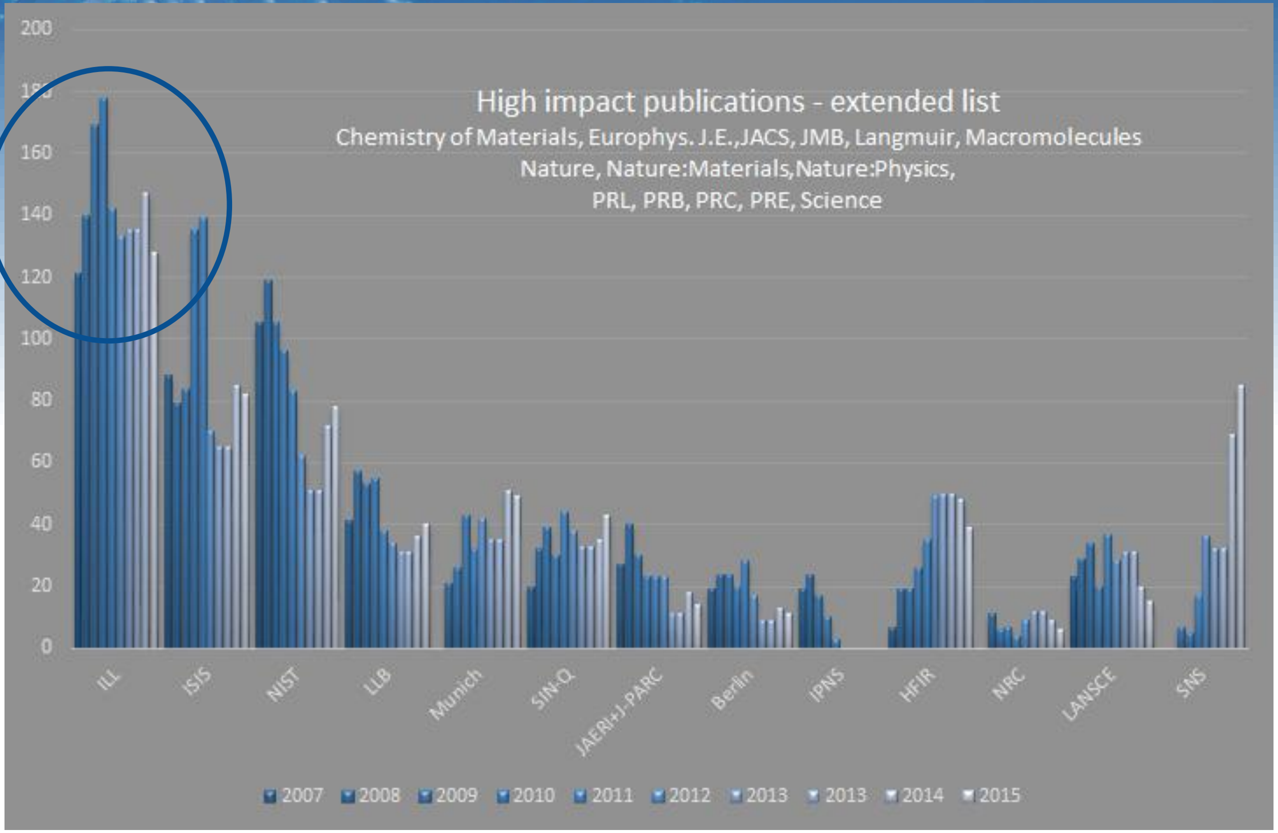
- Stable on the scale of hours up to 140 K
- At 0.81 gm/cm^3 ice XVI constitutes the lightest stable ice phase discovered so far
- Negative thermal expansion for T < 55K

ILL: scientific publications



For 2015: 556 publications (so far),
plus theses, reports, technical documents …

ILL: scientific publications

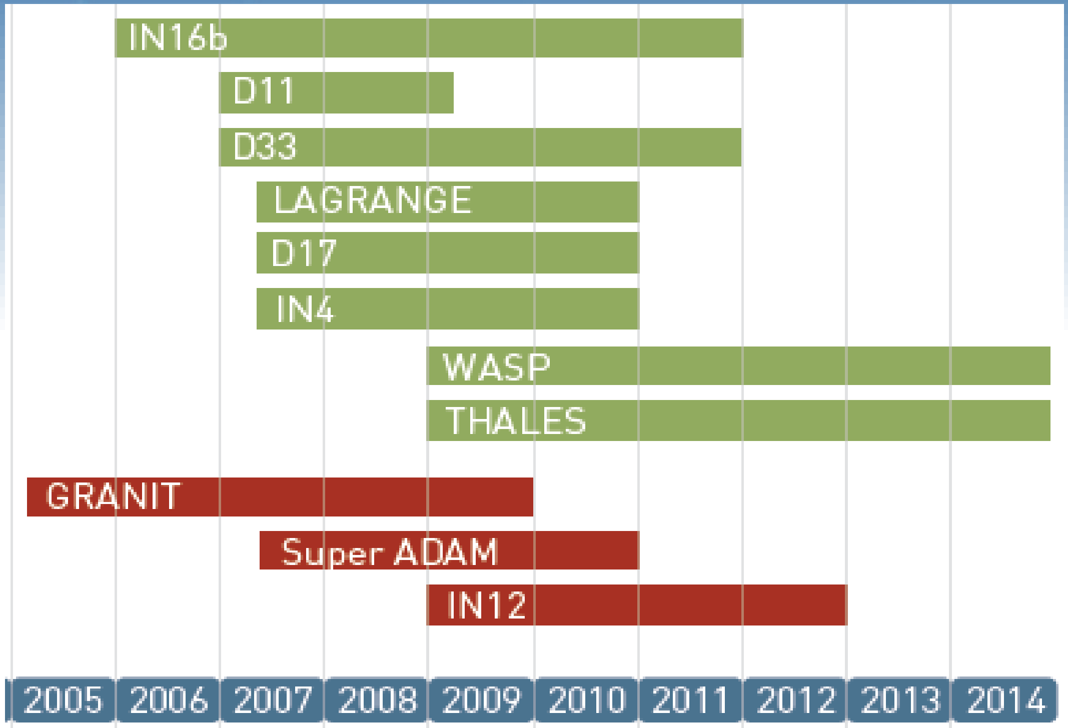


The Future

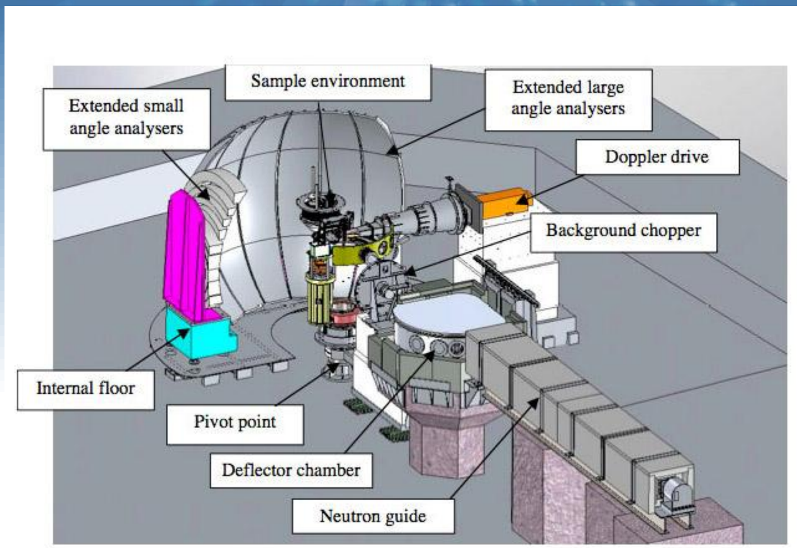
ILL’s upgrade programmes: Millennium and Endurance

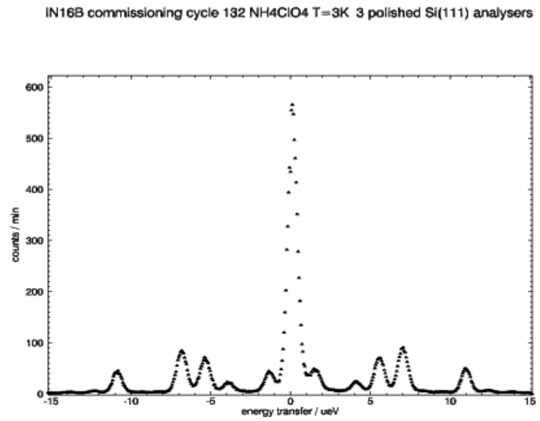
The Millennium Programme

- 2005 – 2016
- constructed or upgraded **14 instruments**
- replaced or renewed a major part of our neutron guides, making them ~ 2 x bright
- Further improved cryostats, magnets, new polarising optics, new electronic instrument control system …



The Millennium Programme
IN16B: backscattering spectrometer



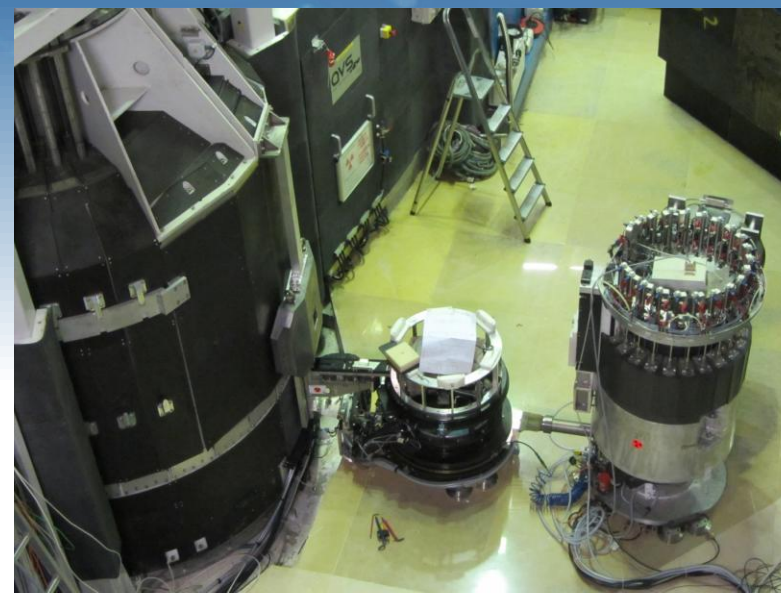


- ➢ Sub-µeV energy resolution
- ➢ Background reduced - analysers and flight path in vacuum (and background chopper)

NEUTRONS FOR SCIENCE®

The Millennium Programme
ThALES: cold-neutron triple-axis spectrometer

Elgoibar, 14/11/2013



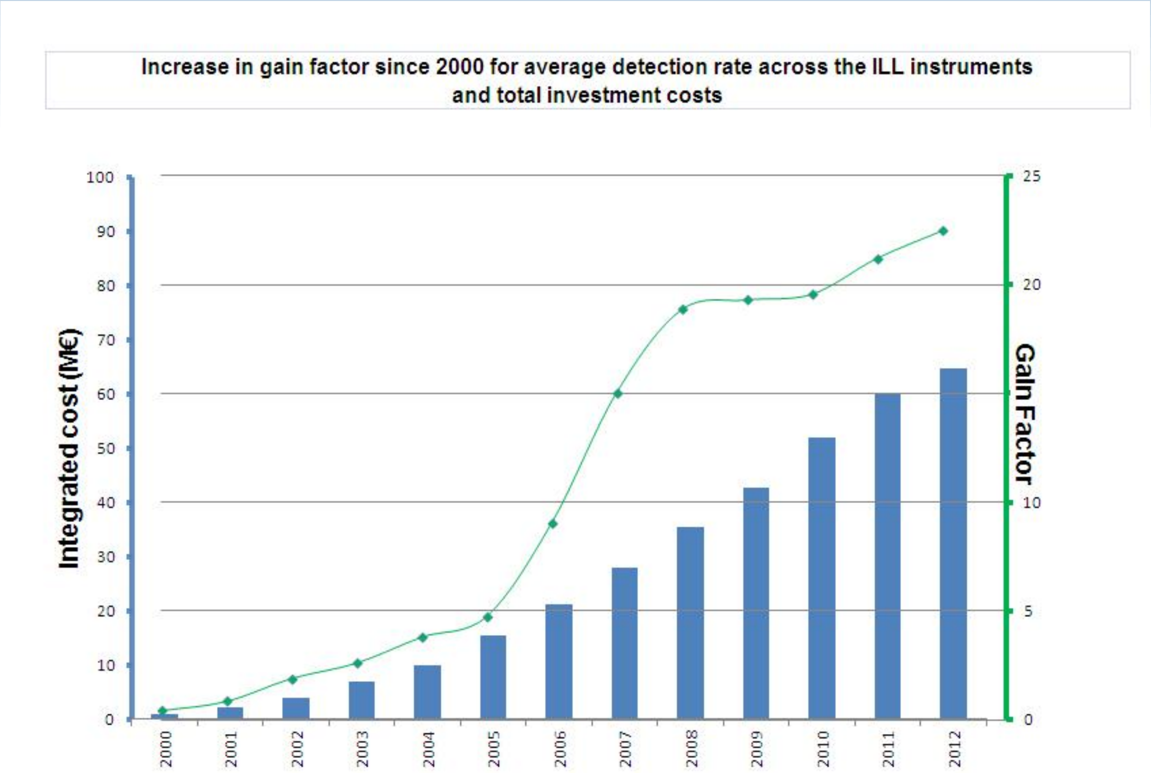
- Flux ≈ 5 x IN14
- H53 end-position
- Nonmagnetic construction
- Polarization analysis

WASP: Neutron Spin Echo

Outstanding possibilities for incoherent scattering (polymers, dynamics of composite materials), diffuse magnetic scattering (e.g. frustrated magnets) …

The Millennium Programme

- Upgrades to instruments, neutron optics …
- Result - the average neutron detection rate on the instruments has been **improved by at least 20**



NEUTRONS FOR SCIENCE®

After Millennium → Endurance

- **Science drivers**: faster measurements, smaller samples, more complex systems
- **Technological development**: higher flux, focusing optics, wider Q range, in single measurement.
- **Sample environment**: particularly for soft condensed matter, biology, during chemical transformations, at extremes of pressure and magnetic field
- **Software**: data reduction and analysis, particularly for non-expert users, developed in partnership

ENDURANCE Programme: Phase 1

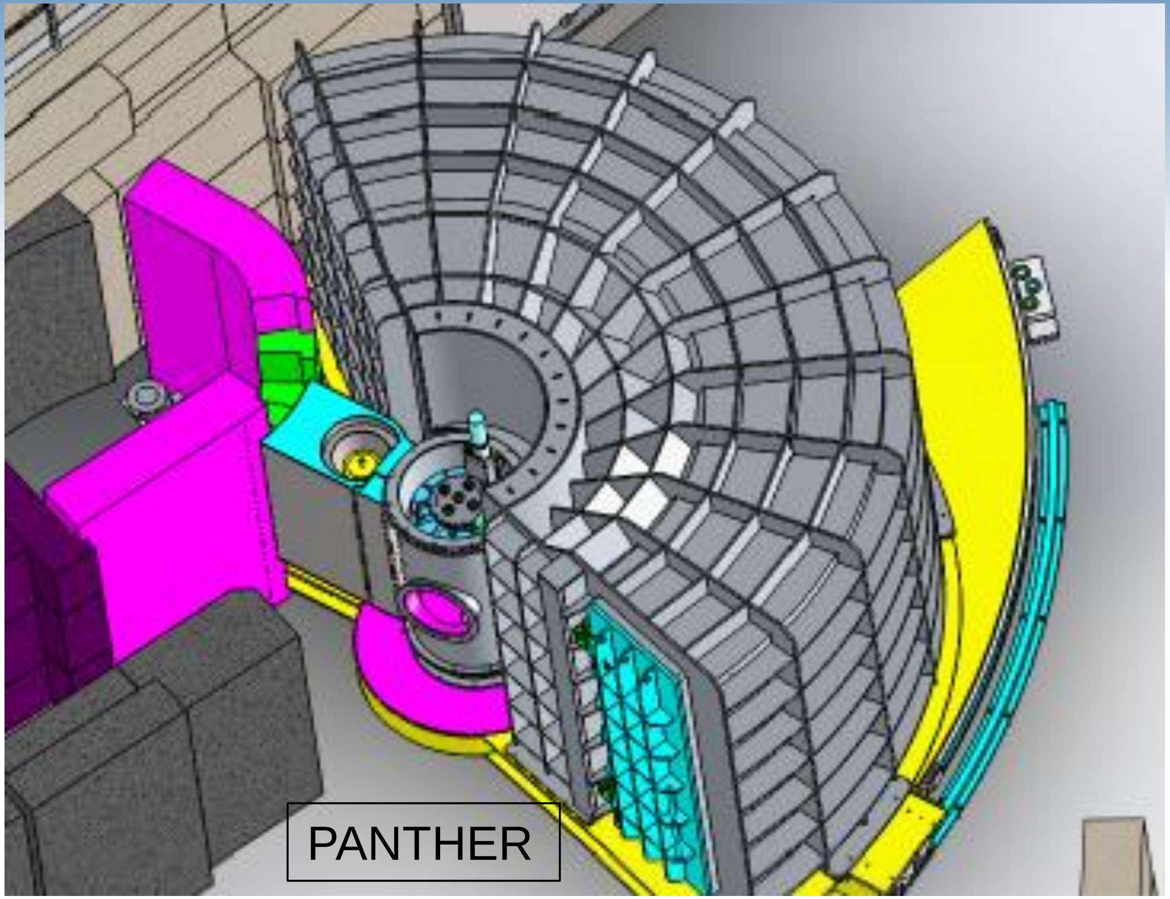
- Instrument projects: SuperSUN, FIPPS, PANTHER, RAINBOWS; 'Chartreuse' projects: XtremeD, D10+, IN13+ (H24)
- Infrastructure projects: H24 neutron guide (H1/H2 in-pile guide)
- Software: BASTILLE (data analysis, with partner institutions; MANTID consortium)
- Sample environment: NESSE (many sub-projects with partners)

- Cost estimate: Phase 1 ~ 22M€

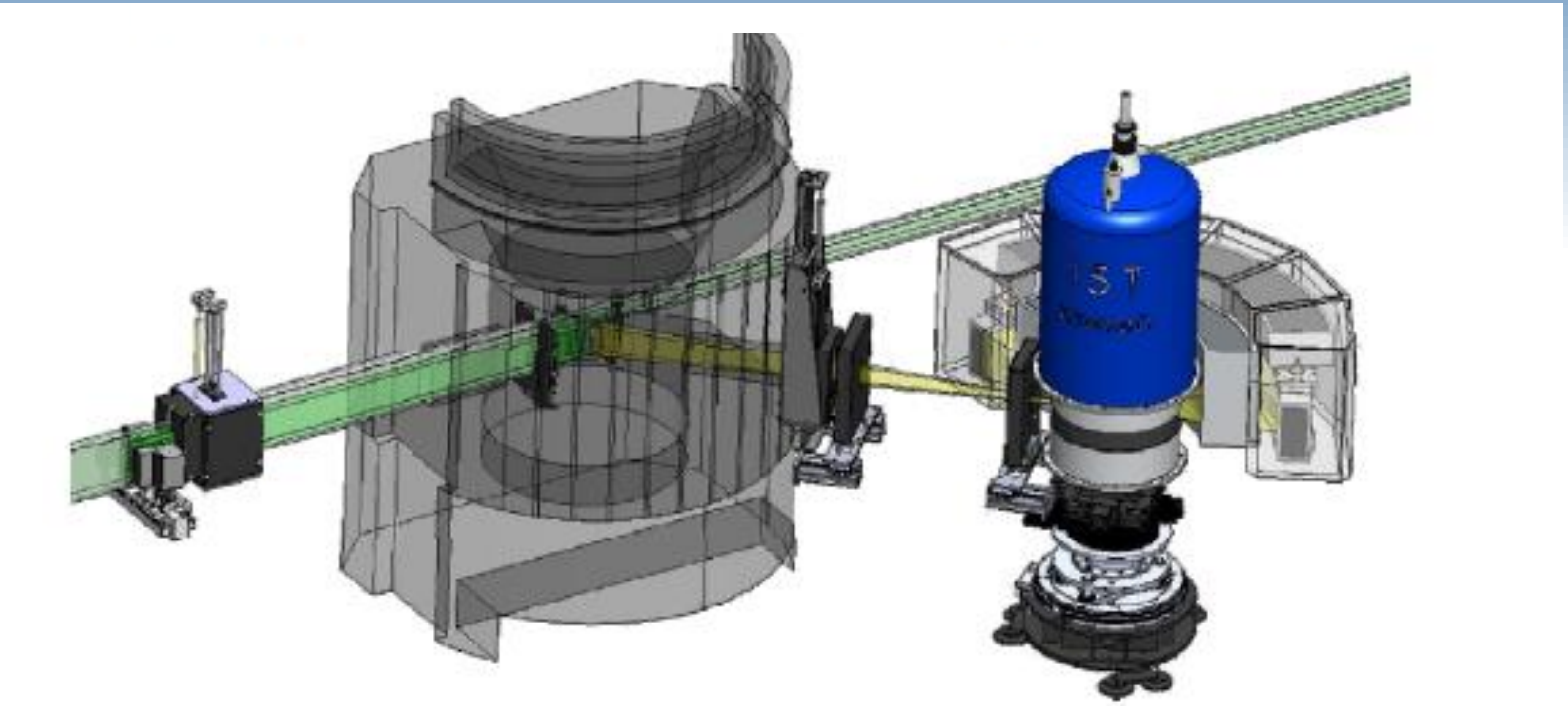
ENDURANCE Programme: Phase 1

➢ XtremeD	Diffractometer, powder and single-crystal, high p and <u>H</u>
➢ FIPPS	Fission product γ spectrometer, exotic nuclei
➢ PANTHER	TOF, thermal, polarisation (IN4)
➢ D10+	3-axis/4-circle, 10 x D10 flux
➢ IN13+	Backscattering, new guide/mono
➢ RAINBOWS	Reflectometry, refractive (prism)
➢ SuperSUN	UCN; n-EDM …
➢ Fa# - IN6	TOF, IN6 upgrade (with LLB)

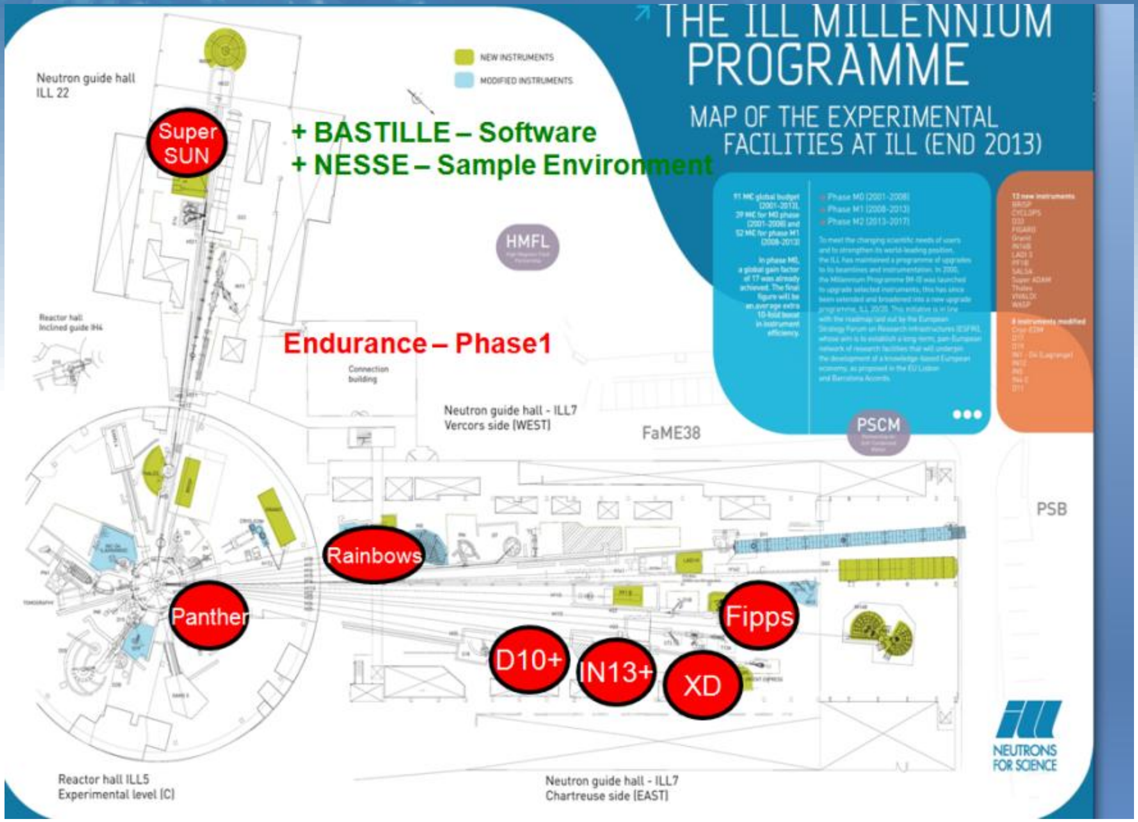
PANTHER: thermal time-of-flight spectrometer with polarised neutrons



XtremeD: diffractometer for powders and single crystals (extreme environments, p and H)



ENDURANCE Programme: Phase 1



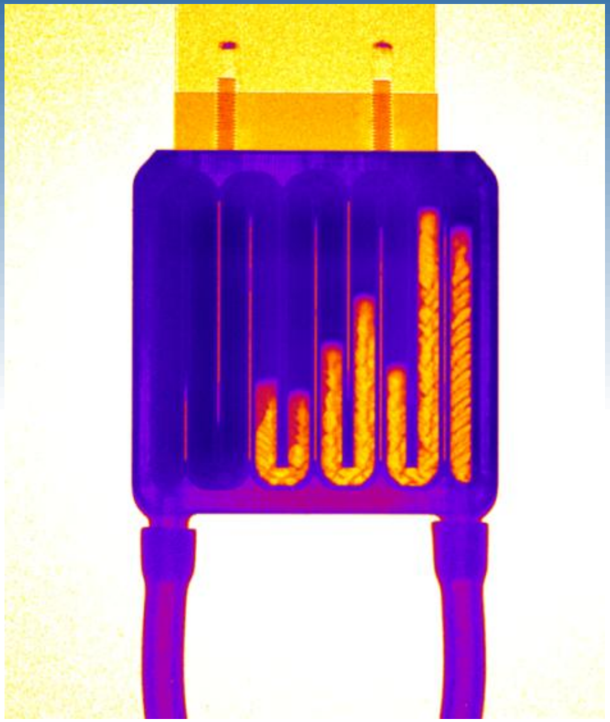
First science: 2017-18 (FIPPS, RAINBOWS, BASTLLE, NESSE)

ENDURANCE Programme: Phase 2

- 2019-2023
- Vercors projet (RAMSES, D7, …); without compromising performance of existing instruments (IN5, D11, FIGARO, D17 …)
- PANTHER phase 2 (monochromator …)
- Continuation of BASTILLE and NESSIE
- Further CRGs?

- Planning underway

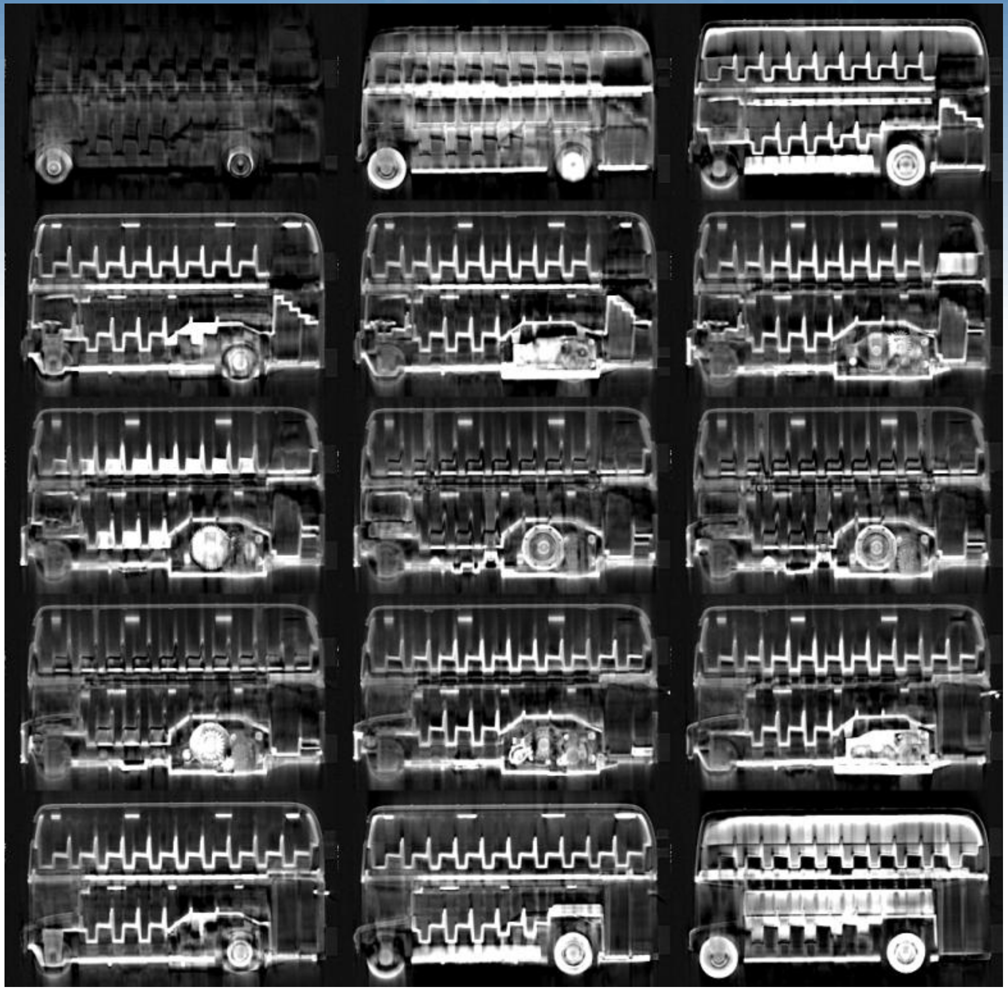
Development of new imaging station on D50



Tenganitti, Ando, Viggiani, Etxegarai,Yehya (UGA/3SR/CNRS), Atkins (ILL): tests, July 2016

3D printed plastic heat exchanger with some water and air trapped in right corner

Development of new imaging station on D50



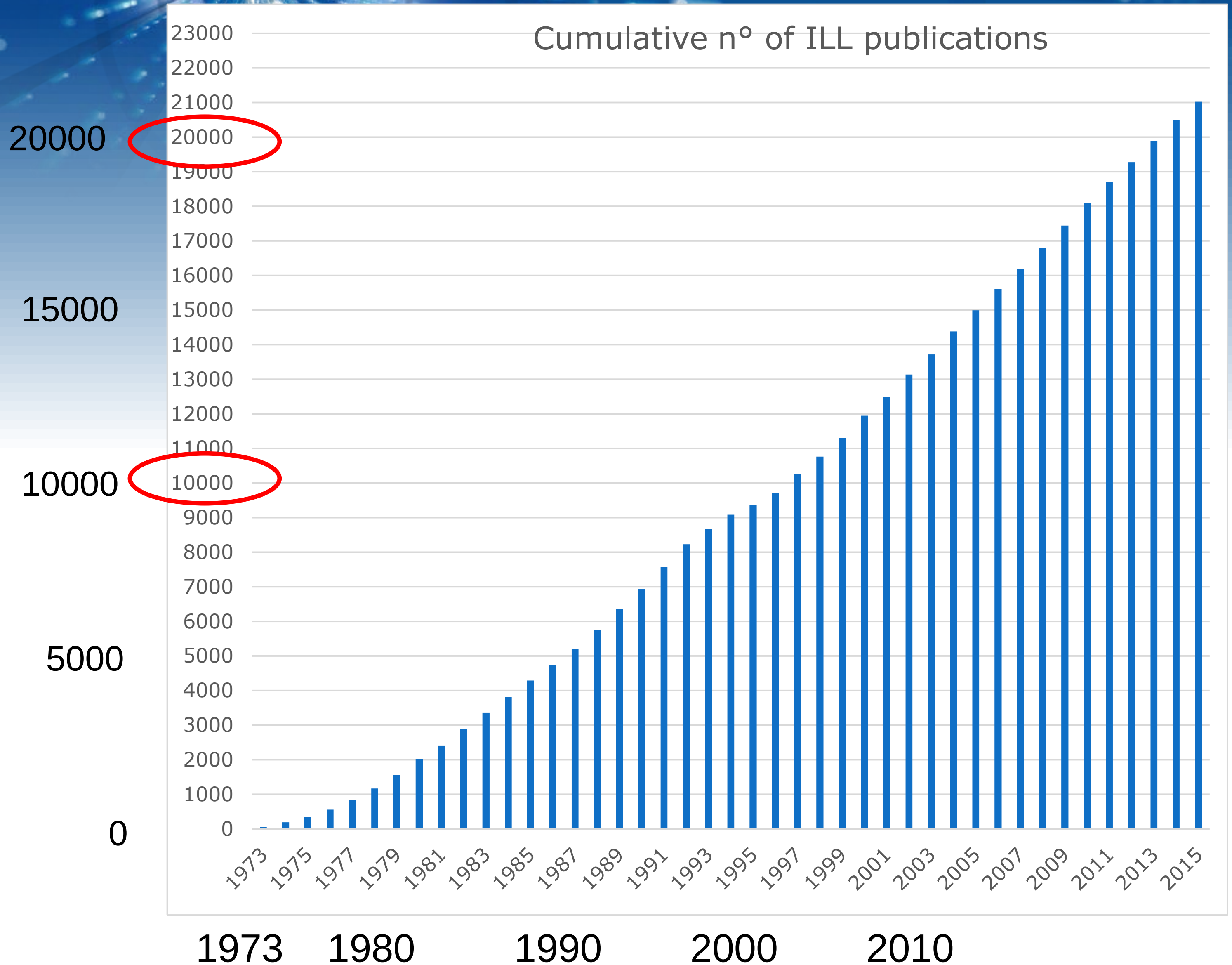
Neutron tomography of London doubledecker - to identify contrast in different materials and test reconstruction software

3D rendering – to be improved

19 January 1967: signature of agreement between France and Germany

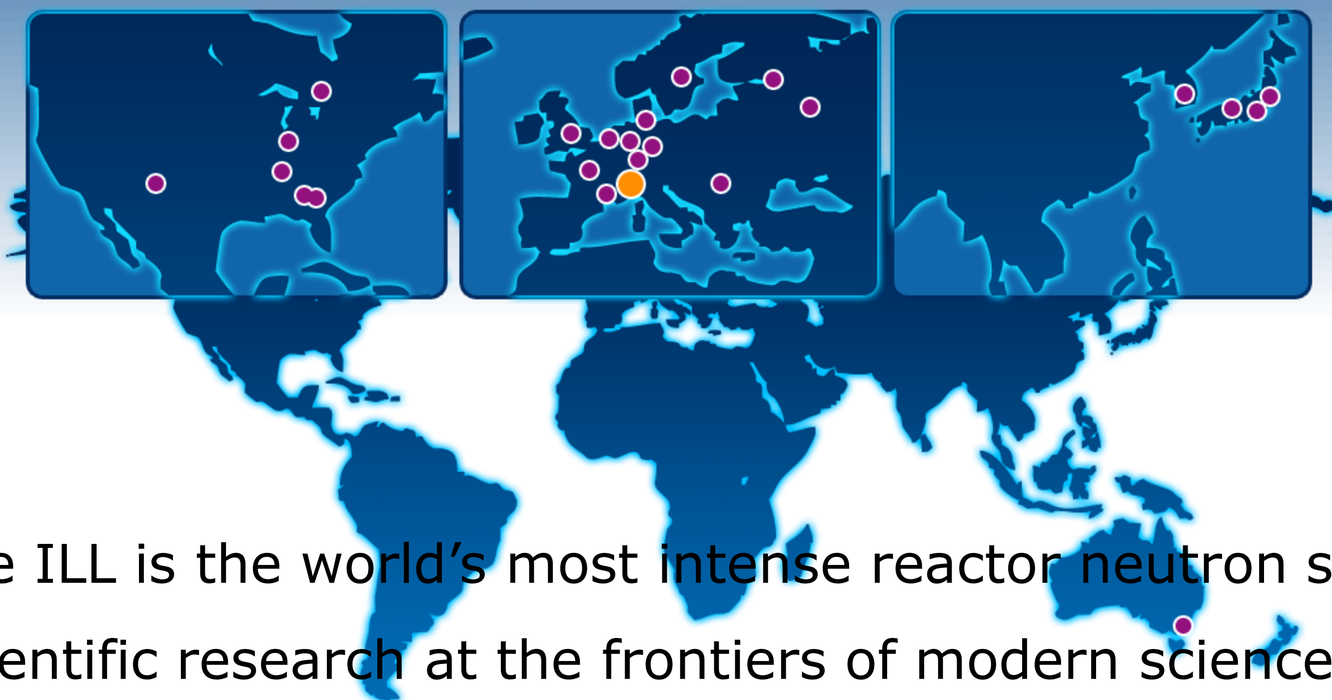
19 January 2017: ILL's 50th anniversary

ILL: scientific publications



Institut Laue-Langevin

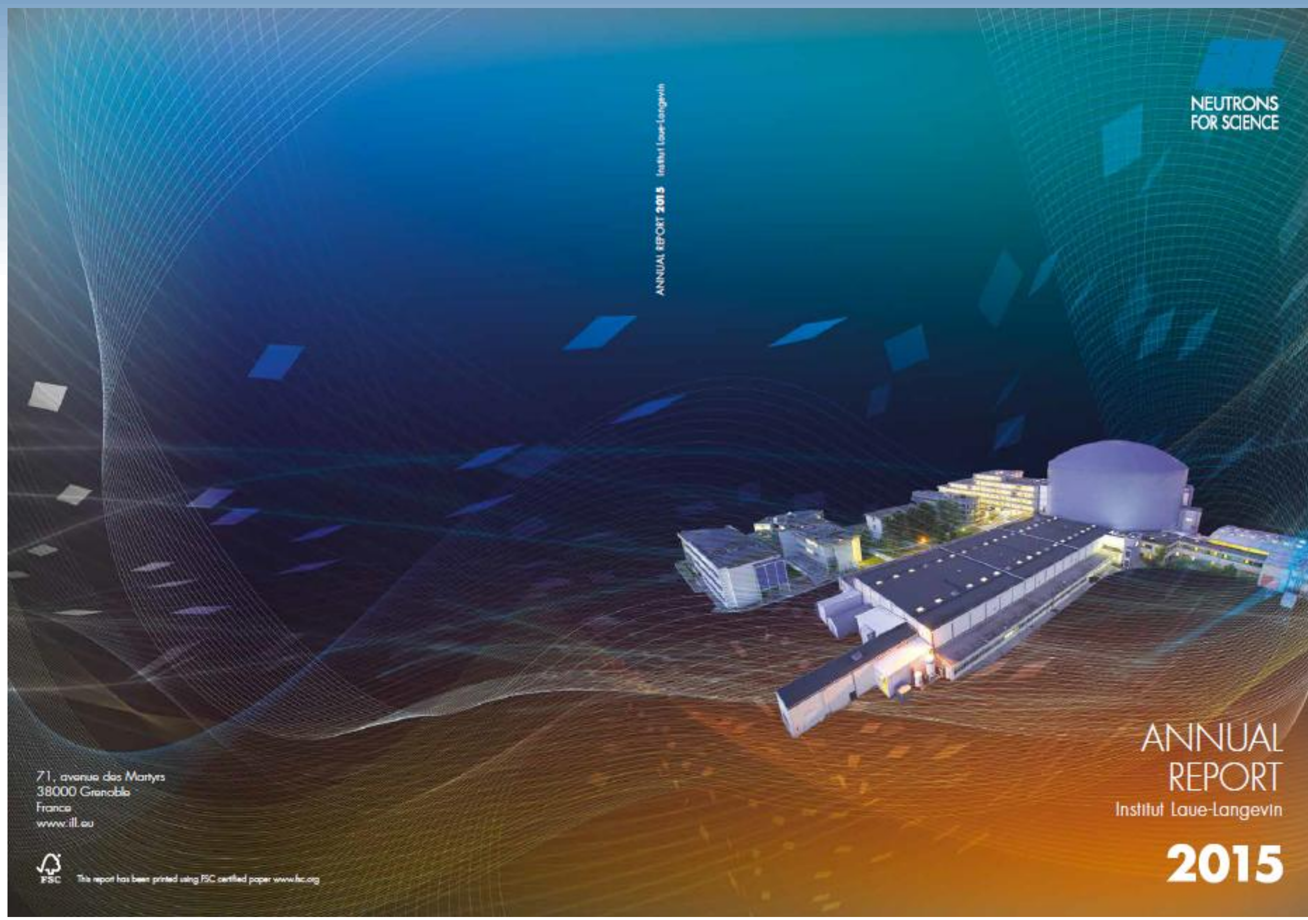
Founded in 1967, world leader in neutron science and technology



- The ILL is the world's most intense reactor neutron source
- Scientific research at the frontiers of modern science; scientific publications, training, technique and instrument development …
- Endurance programme maintains world-leading position

NEUTRONS
FOR SCIENCE ®

Annual report 2015



NEUTRONS
FOR SOCIETY

Thank you for your attention