

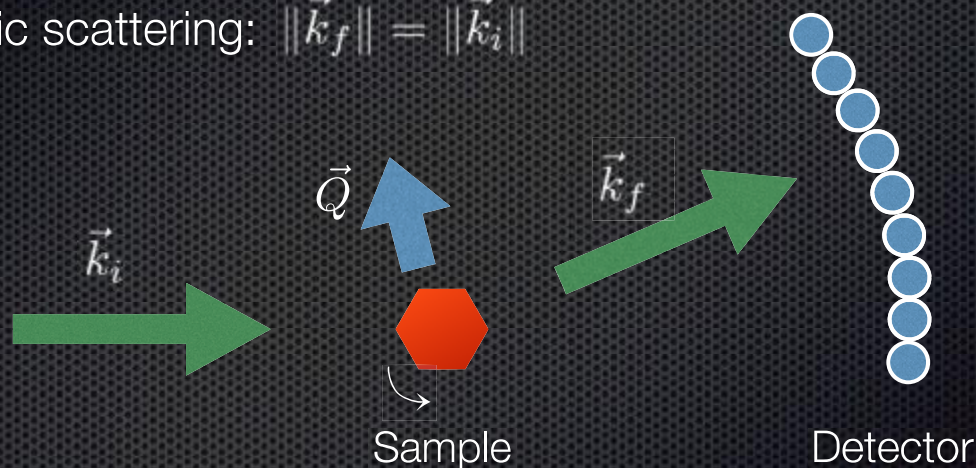
Neutron Instrumentation

Eddy Lelièvre-Berna — lelievre@ill.eu

- ❖ What do we measure and need ?
 - ❖ Neutron source and guides
 - ❖ Measuring techniques
 - ❖ Energy selectors and polarisers
 - ❖ Sample environments
 - ❖ Neutrons detectors
 - ❖ Data acquisition system

What do we measure ?

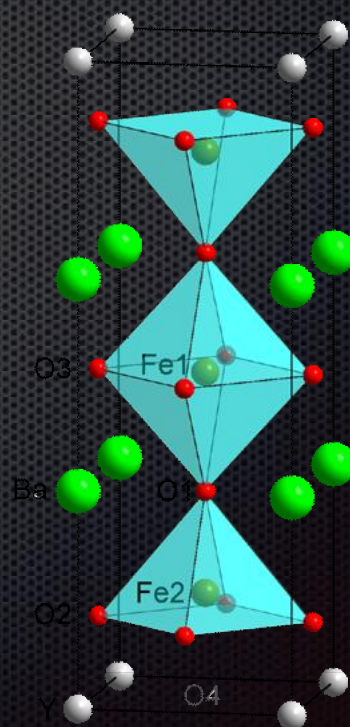
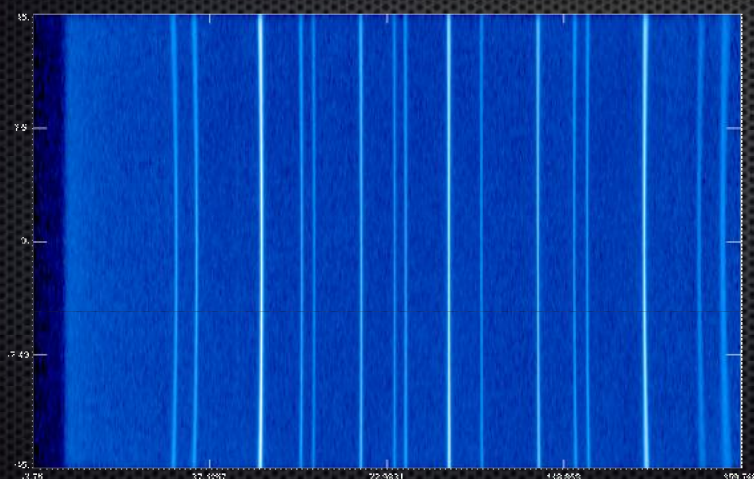
- Elastic scattering: $\|\vec{k}_f\| = \|\vec{k}_i\|$



Intensity vs wavevector transfer $\vec{Q} = \vec{k}_f - \vec{k}_i$

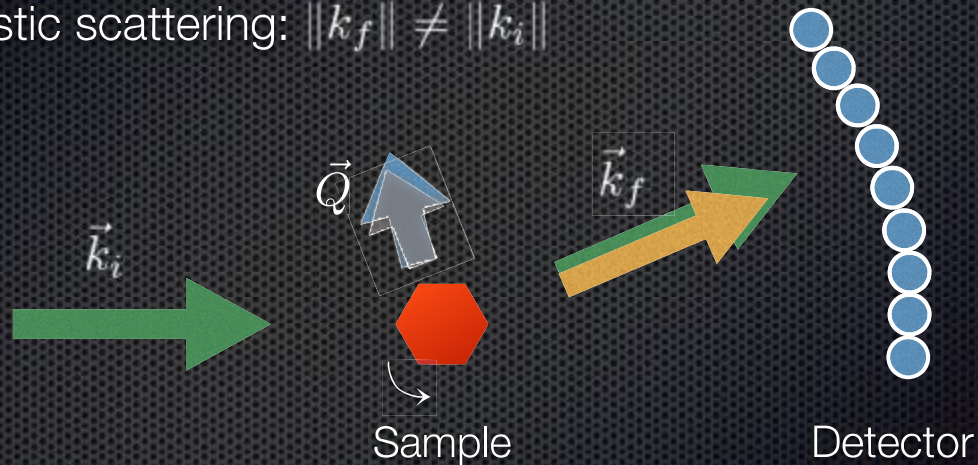
What do we measure ?

- Example: $\text{YBa}_2\text{Fe}_3\text{O}_8$ structure



What do we measure ?

- Inelastic scattering: $\|\vec{k}_f\| \neq \|\vec{k}_i\|$



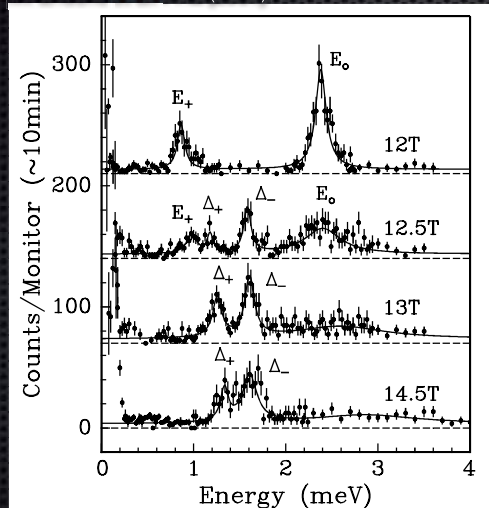
Intensity vs wavevector and energy transfers

$$\vec{Q} = \vec{k}_f - \vec{k}_i, \quad \hbar\omega = E_f - E_i$$

What do we measure ?

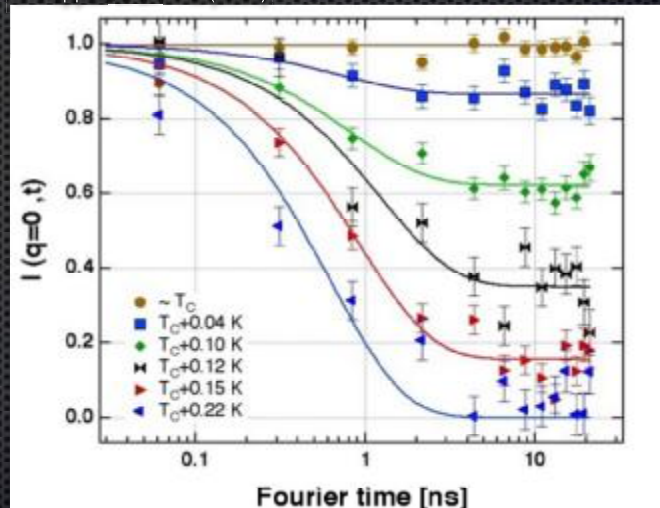
- Examples: correlated motions, relaxation

M. Enderle *et al.* PRL (2001)



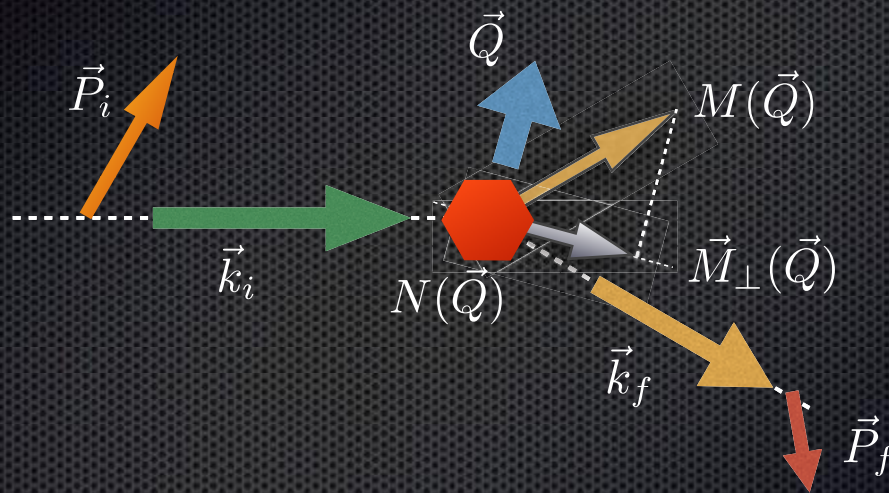
Excitations in CuGeO_3

C. Pappas *et al.* PRL (2009)



Skyrmions in MnSi

What do we measure ?



In general, the polarisation of a neutron beam will change both in magnitude and direction upon scattering from a magnetic material.

What do we measure ?

- Polarised neutron scattering (elastic/inelastic):
 - We measure spin-dependent intensities:

$$I_{+,+}, I_{-,+}, I_{+,-}, I_{-,-}$$

- and components of the scattered polarisation \vec{P}_f for each direction of the incident polarisation \vec{P}_i :

$$P_{i,j} = \frac{P_i P_{i,j} + P_j^\dagger}{\|\vec{P}_f\|} \text{ with } (i,j) \in \{x,y,z\}$$

So what do we need ?

- Source of neutrons
- One or two wavelengths
 - ↳ Monochromators, choppers, Larmor labelling, etc.
- Incident and scattered neutron directions
 - ↳ Collimations, encoded shafts, Tanzboden, slits, etc.
- Incident (and scattered) polarisations
 - ↳ Monochromators, supermirrors, spin filters & flippers
- Monitors and detectors

- What do we measure and need ?
 - Neutron source and guides
 - Measuring techniques
 - Energy selectors and polarisers
 - Sample environments
 - Neutrons detectors
 - Data acquisition system

Neutron sources



1943 — Oak Ridge National Lab. (USA)

The Clinton Pile was the world's first continuously operated nuclear reactor.

Neutron sources



2013 — Oak Ridge National Lab. (USA)

1.4 MW Spallation Neutron Source

Neutron sources



2023 — European Spallation Source (Sweden)
5 MW long pulse source — 1.8 Billion €

Neutron sources



2023 — European Spallation Source (Sweden)
5 MW long pulse source — 1.8 Billion €

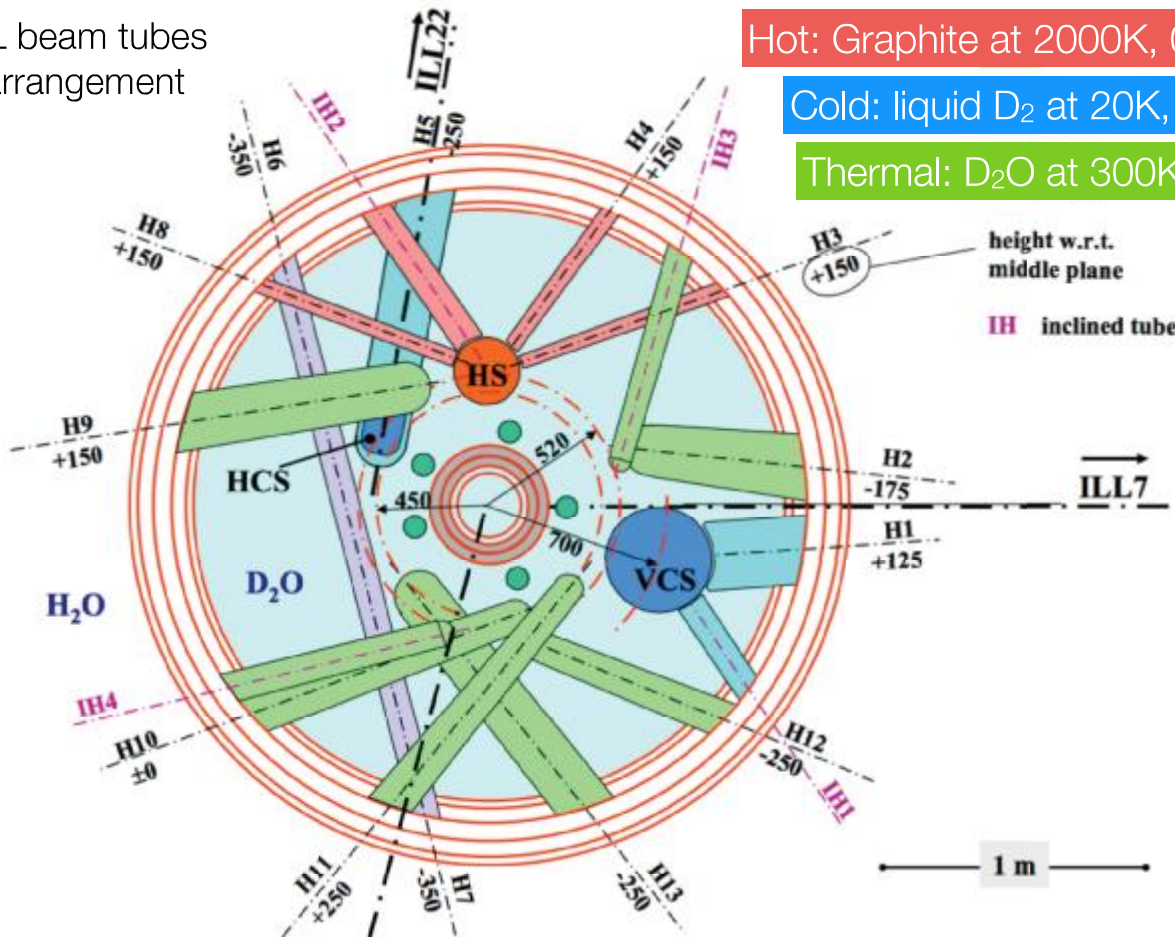
Neutron sources



≈ 50 Centres
≈ 30 Sources

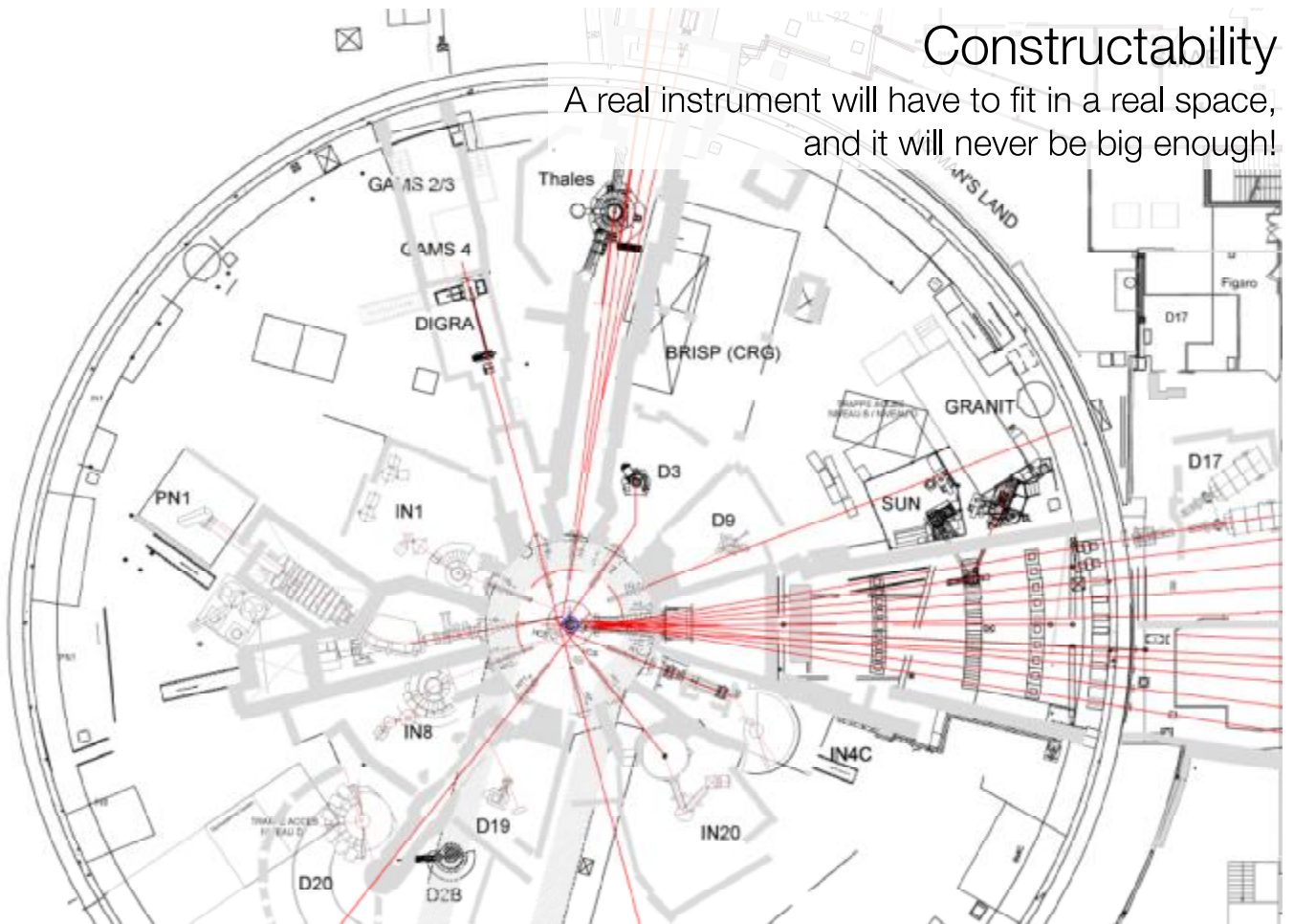


ILL beam tubes arrangement



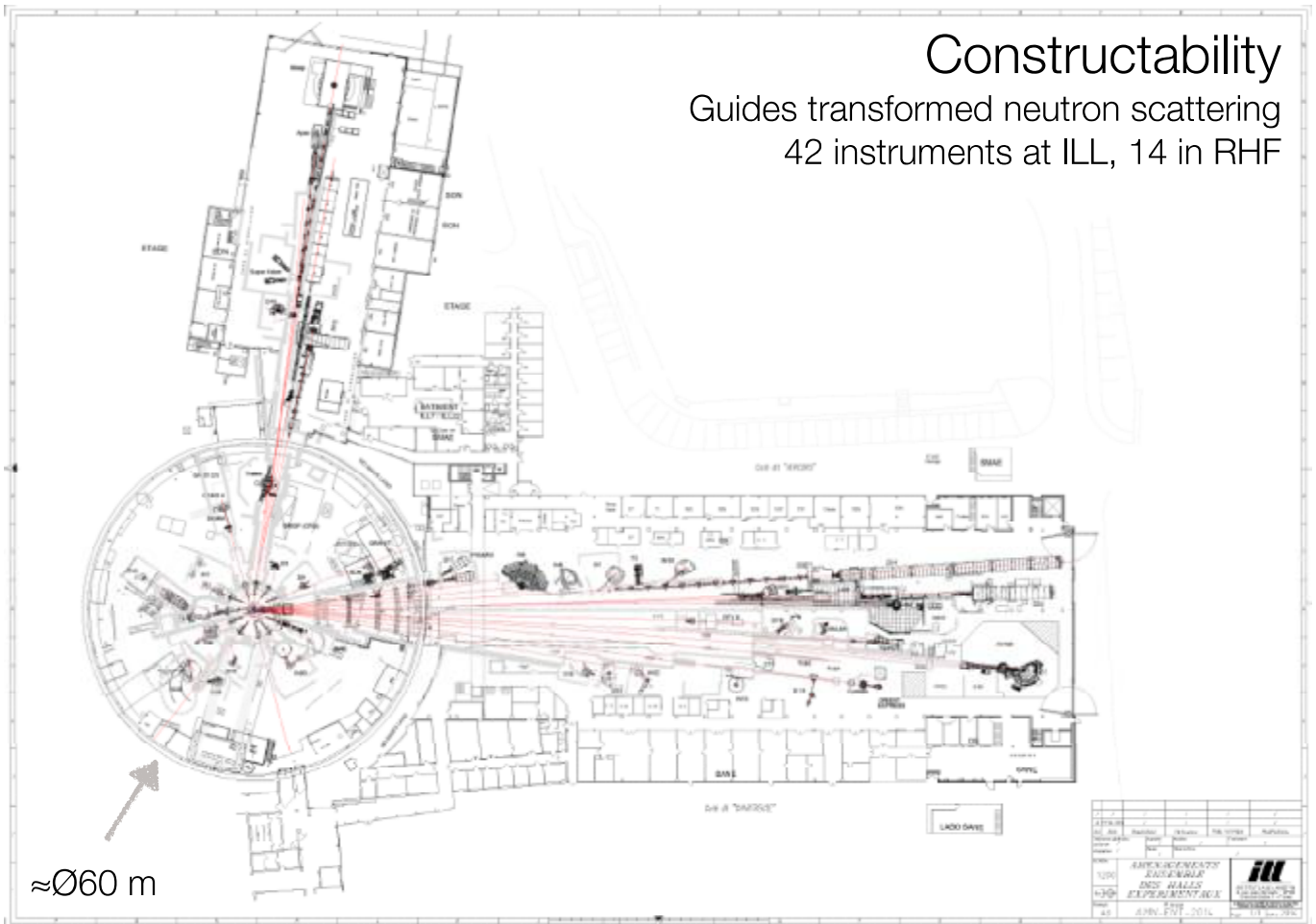
Constructability

A real instrument will have to fit in a real space, and it will never be big enough!



Constructability

Guides transformed neutron scattering
42 instruments at ILL, 14 in RHF

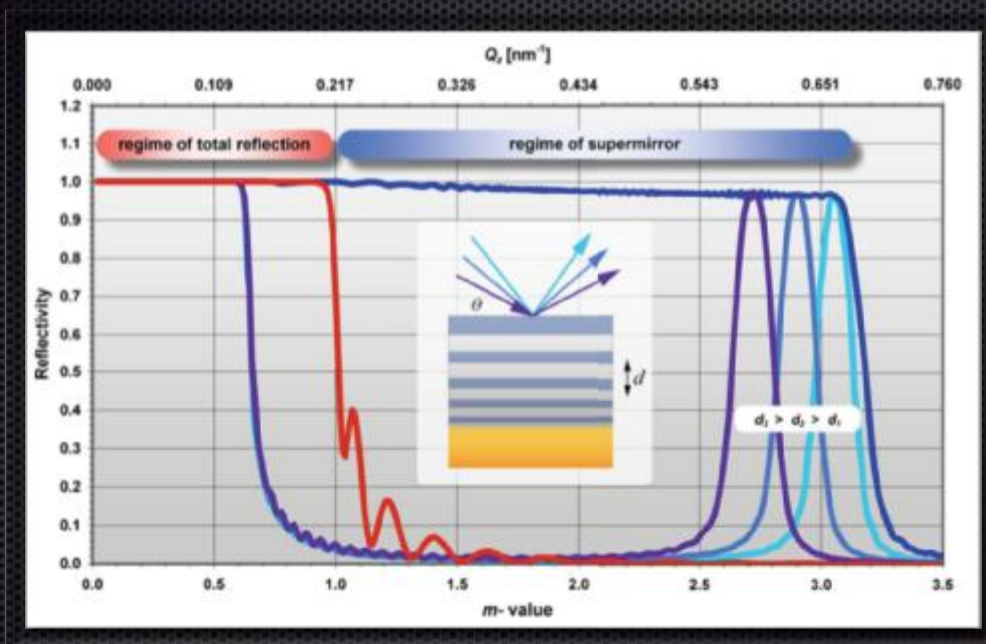


Supermirror neutron guides

- A guide is made up of sections joined together
- Glass is sufficiently thick to hold the vacuum
- Curved guides can eliminate fast neutrons
- Guides can focus, collimate, polarise...



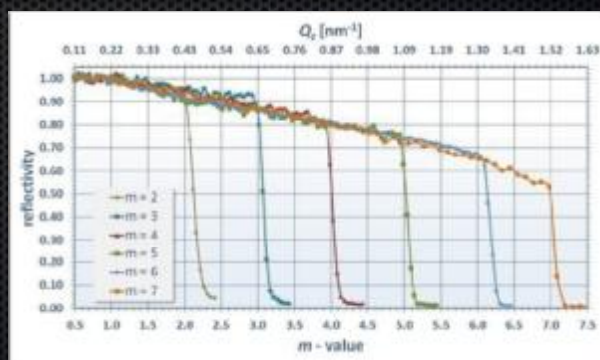
Supermirror neutron guides



$$m = \theta_c / \theta_c^{Ni}, \quad \theta_c^{Ni} \approx 0.1^\circ / \text{\AA}$$



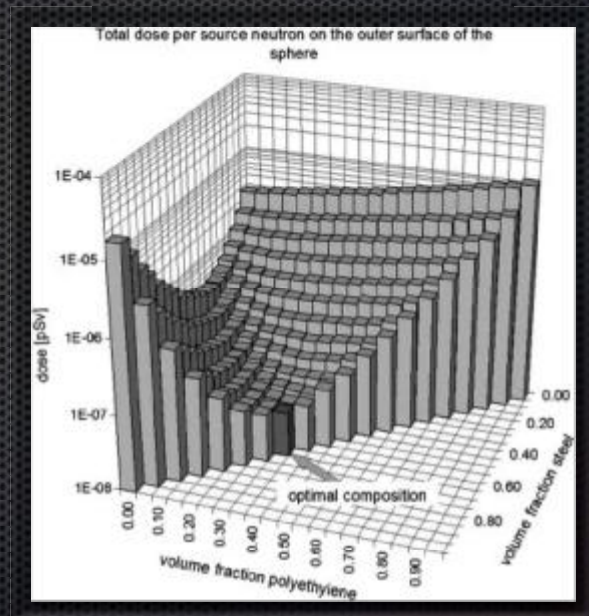
H5 installation in 2014 at ILL



Swiss Neutronics

Shield against fast neutrons, thermal neutrons, gammas

- ✦ Hydrogeneous: concrete, wax, polyethylene
- ✦ Lead, Iron (steel)
- ✦ Boron, ${}^6\text{Li}$, Cd, Gd/GdO
- ✦ We always need better shielding...



Calzada *et al.* NIMA 651 (2011) 77

- ✦ What do we measure and need ?
 - ✦ Neutron source and guides
 - ✦ Measuring techniques
 - ✦ Energy selectors and polarisers
 - ✦ Sample environments
 - ✦ Neutrons detectors
 - ✦ Data acquisition system

Introduction



In 1944, Ernest O. Wollan and Lyle B. Borst performed pioneering neutron diffraction experiments with a modified X-ray diffractometer.

Introduction



Ernest O. Wollan & Clifford G. Shull taking data on a double-crystal neutron spectrometer at the ORNL X-10 graphite reactor in 1949.

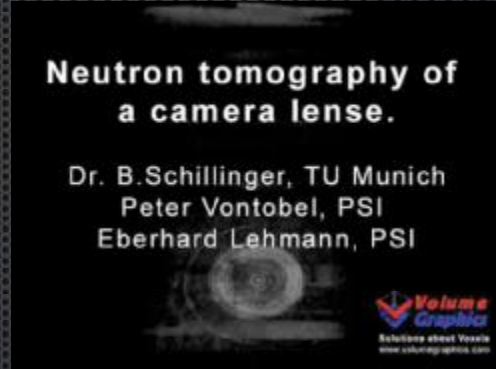
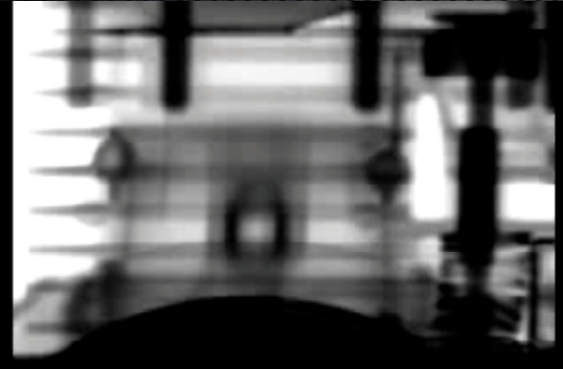
Measuring techniques

- Neutronography
- Diffraction (elastic scattering)
 - Powder diffraction: low and high resolutions
 - Single crystal diffraction: normal-beam, 4-circle, Laue
 - Small angle scattering
 - Reflectometry (horizontal, vertical)

Measuring techniques

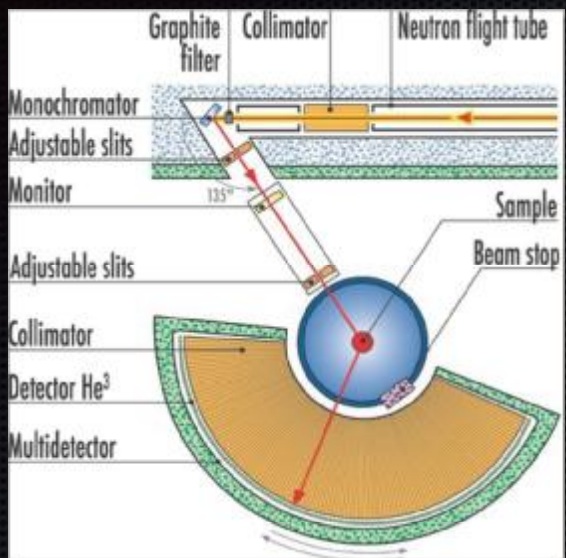
- Spectroscopy (inelastic scattering):
 - Three-axis: hot, thermal and cold energies (\approx meV)
 - Time-of-flight (\approx 0.1 meV)
 - Backscattering (\approx μ eV)
 - Spin-echo: std/para/ferro, etc. (\approx 0.2 neV to 200 μ eV)
- Nuclear & particle physics instruments

Measuring techniques

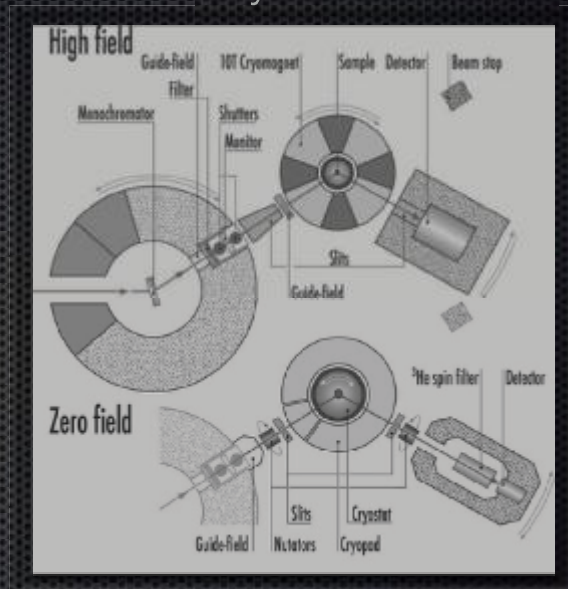


5 μm resolution available — complementary to x-rays

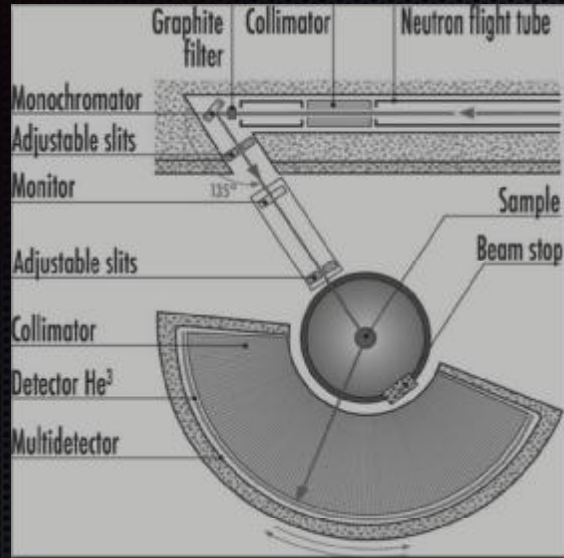
Powder diffraction



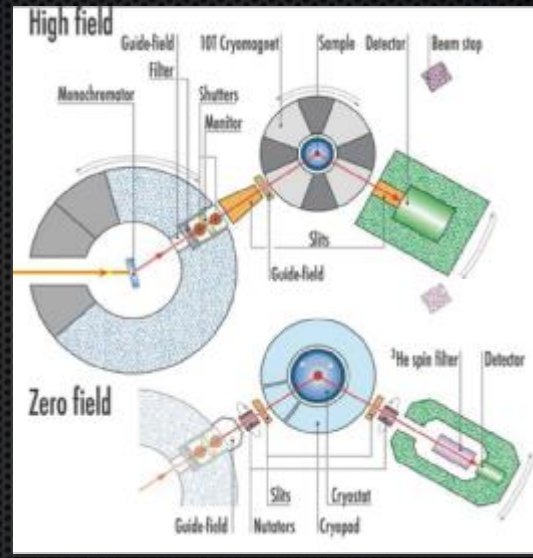
Crystal diffraction



Powder diffraction

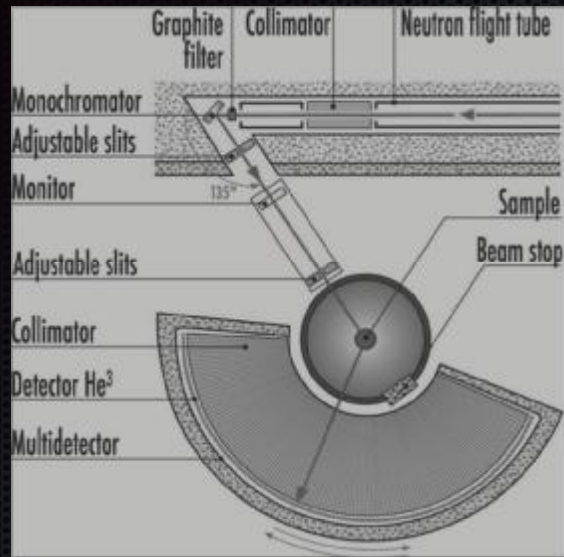


Crystal diffraction

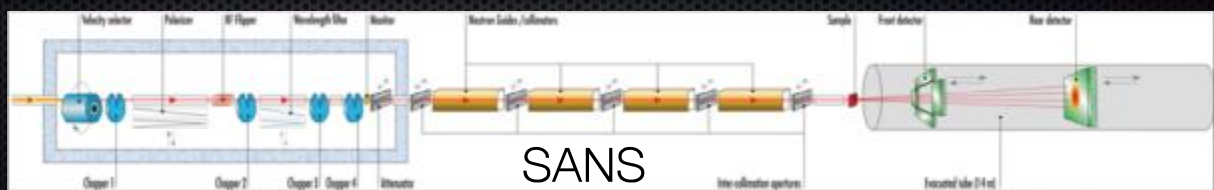
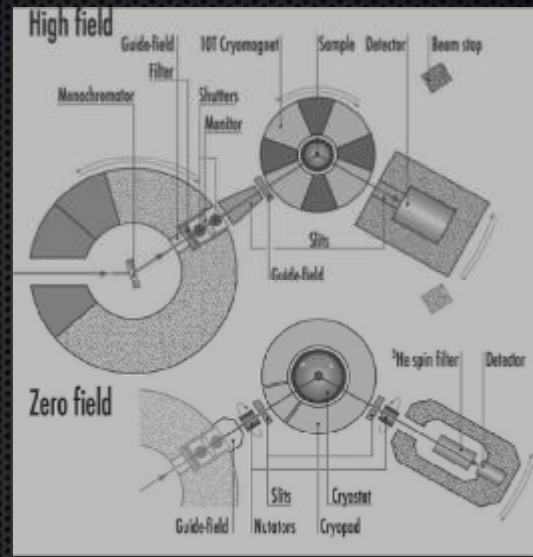


SANS

Powder diffraction

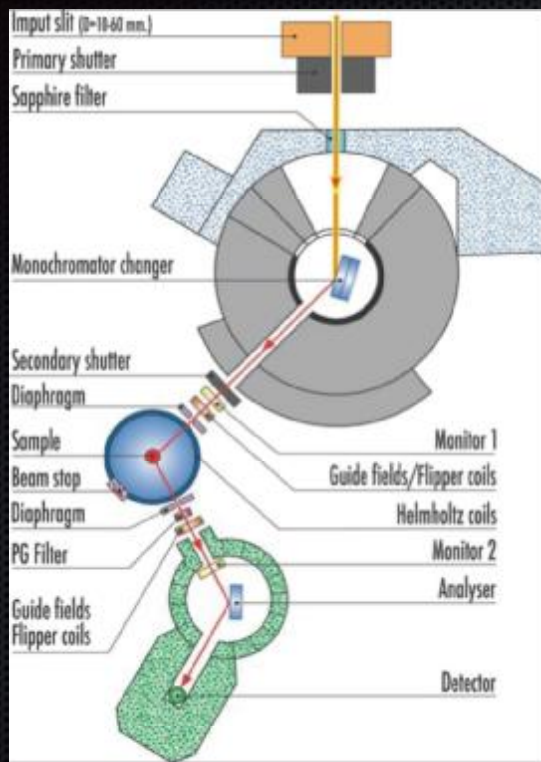


Crystal diffraction

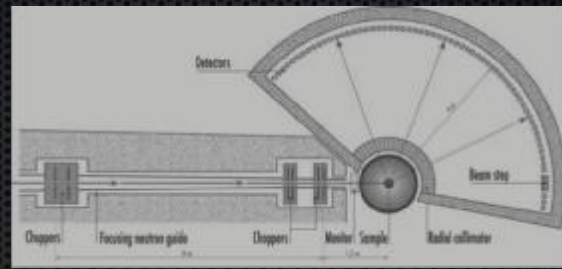


SANS

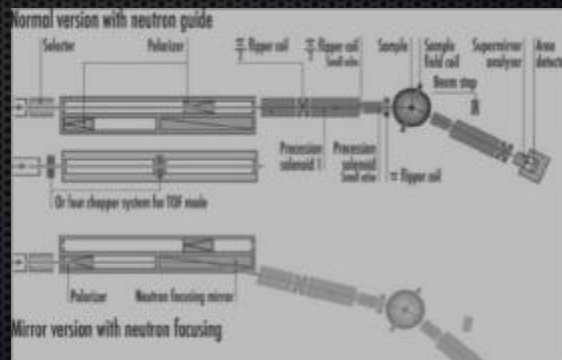
Three-axis



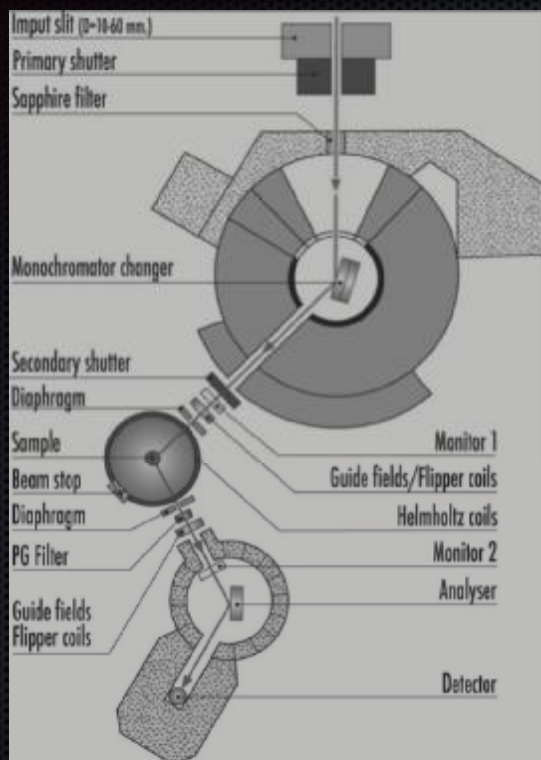
Time of flight



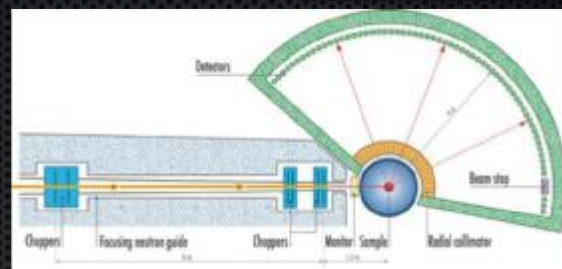
Spin-echo



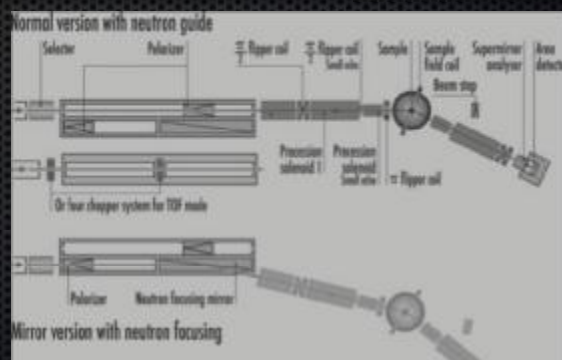
Three-axis



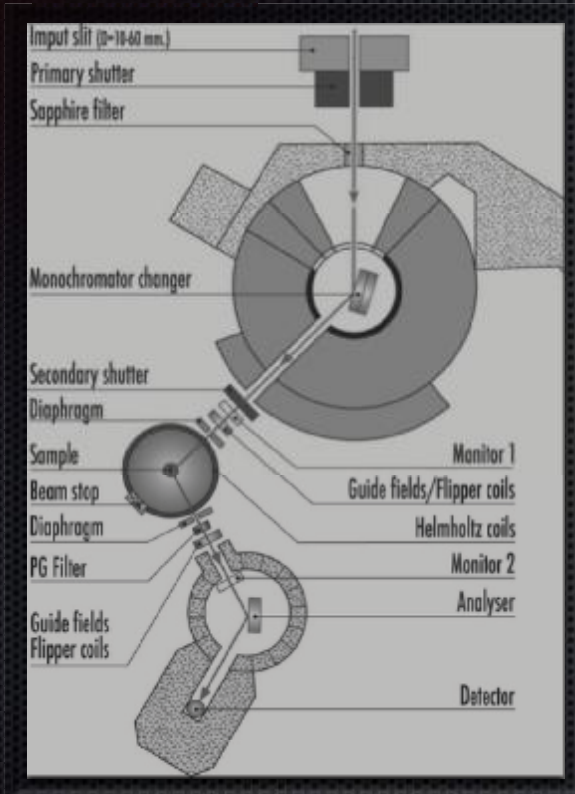
Time of flight



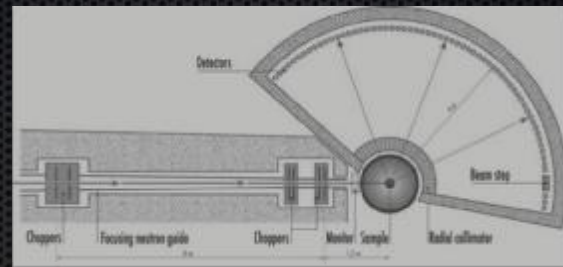
Spin-echo



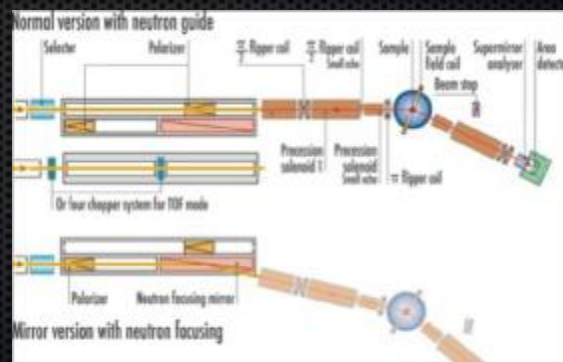
Three-axis



Time of flight



Spin-echo



- ❖ What do we measure and need ?
 - ❖ Neutron source and guides
 - ❖ Measuring techniques
 - ❖ Energy selectors and polarisers
 - ❖ Sample environments
 - ❖ Neutrons detectors
 - ❖ Data acquisition system

Energy selectors & polarisers

- Energy selection
 - Monochromators
 - Time-of-flight selection devices
- Spin selection, manipulation
 - Polarising crystals, supermirrors, ^3He spin filters, etc.
- Removal of unwanted energies and neutrons
 - Filters (e.g. Be, MgO), (oscillating) collimators, slits, etc.

Monochromators

- Array of single crystals
 - To select energy (and polarisation)
 - Cu, Si, HOPG, Heusler, Diamond...
 - Flat or focusing vertically (and horizontally)
 - Optimised mosaic



Bridgman furnace at ILL

Monochromators



Heusler (Cu_2MnAl) polarising crystal (ILL)



$\text{\O}80 \times 300 \text{ mm}^3$ Cu crystal (ILL)

Monochromators



x5 flux @ 1.5 \AA

IN8 doubly-focusing monochromator



x2 flux @ 1.3 \AA

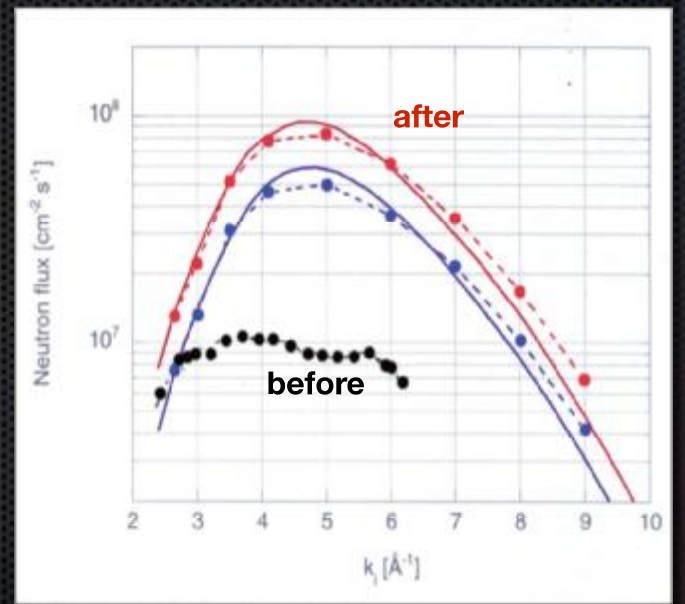
D20 Cu monochromator

Monochromators

IN20 Monochromator



75 Heusler crystals

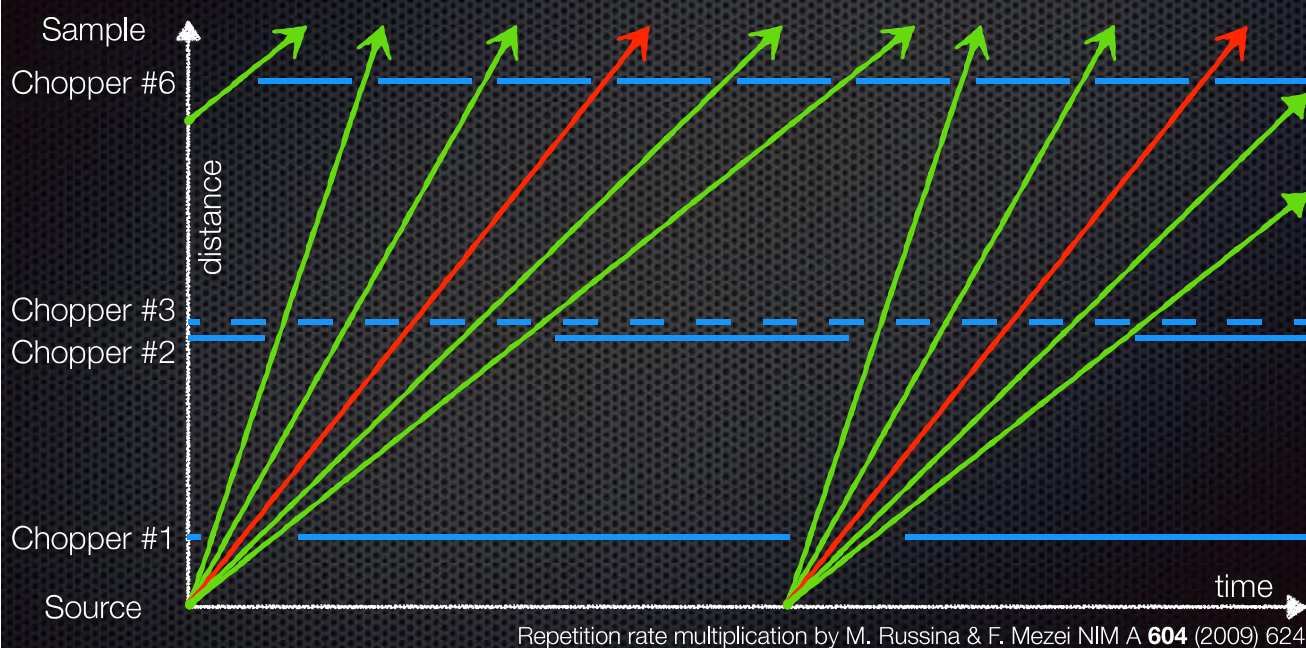


92% polarisation, x10 flux (from Kulda *et al.*)

Time of Flight devices

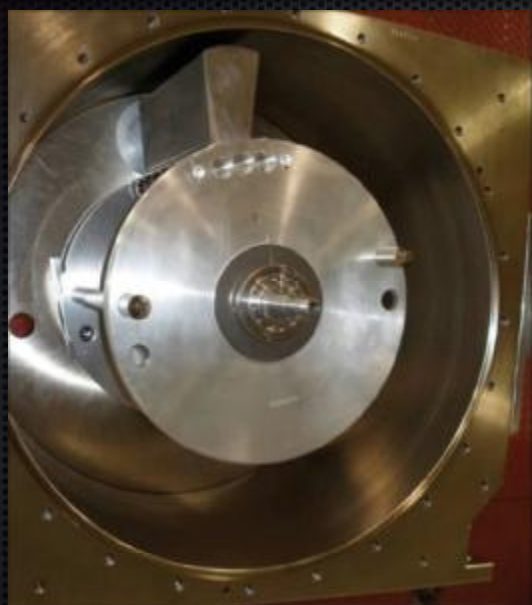
- ✦ Disk choppers
 - ✦ T_0 choppers to stop fast neutrons (pulsed sources)
 - ✦ Bandwidth-limiting choppers (prevent frame overlap)
 - ✦ E_0 or Fermi choppers to transmit a very narrow bandwidth of neutrons (e.g. to define E_i)
- ✦ Velocity selectors

Time of Flight technique



T₀ choppers

Courtesy to I. Anderson



Single-blade rotor



Assembled chopper unit

Bandwidth-limiting choppers

Courtesy to I. Anderson



60 Hz chopper disk (TOPAZ, SNS)



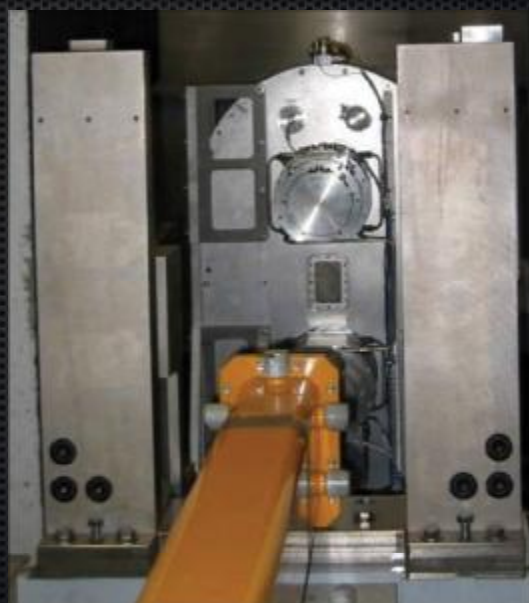
Installed on beam line

Bandwidth-limiting choppers

Courtesy to I. Anderson



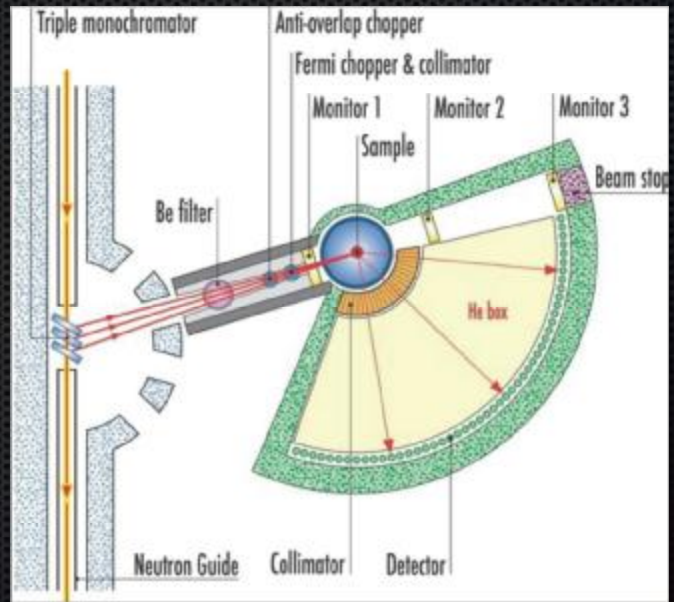
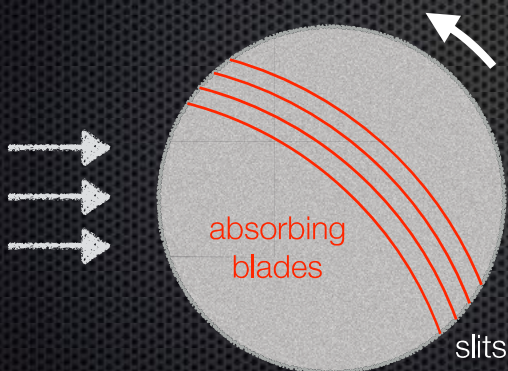
300 Hz dual counter-rotating disks



Installed on beam line

Fermi choppers

- ToF focusing
- Monochromator



IN6 layout

Velocity selectors

- Large $\Delta\lambda/\lambda$, typically 10% resolution
- High transmission, between 5 and 30%
- Typically 1.000 rpm
- Multi-disc or multi-blade

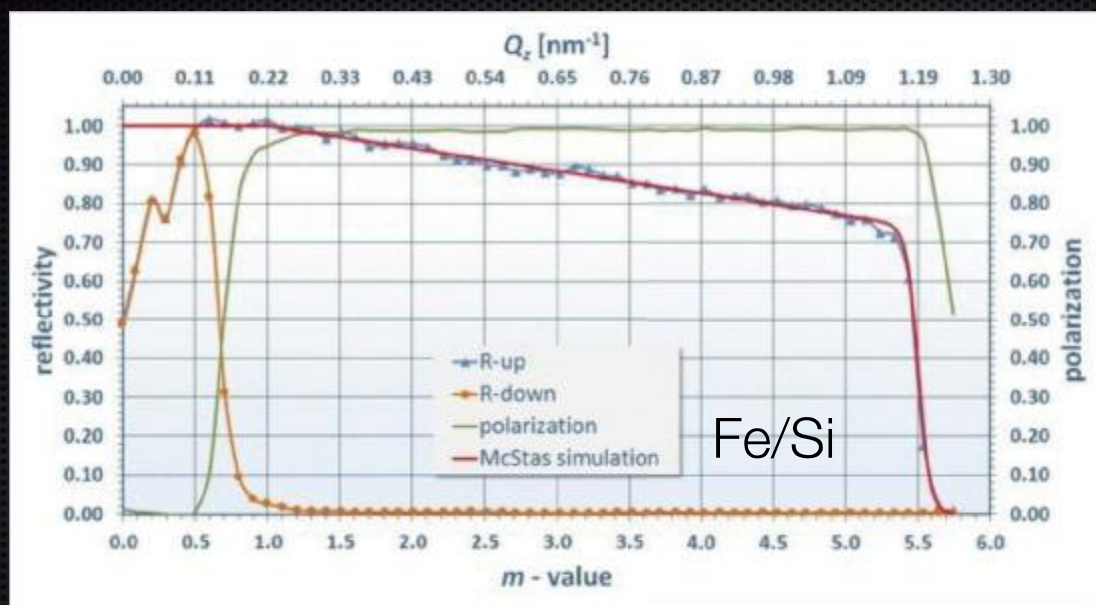


Astrium GmbH

Spin selection, manipulation

- The polarisation can be seen as a vector in space
- Polarisers & spin analysers:
 - Heusler crystals, supermirrors, ^3He spin filters
- Spin flippers: Mezei, Tasset, RF adiabatic, etc.
- Zero-field polarimeters: Cryopad, MuPad

Polarising supermirrors



Swiss Neutronics

$$m = \theta_c / \theta_c^{Ni}, \quad \theta_c^{Ni} \approx 0.1^\circ / \text{\AA}$$

Polarising supermirrors



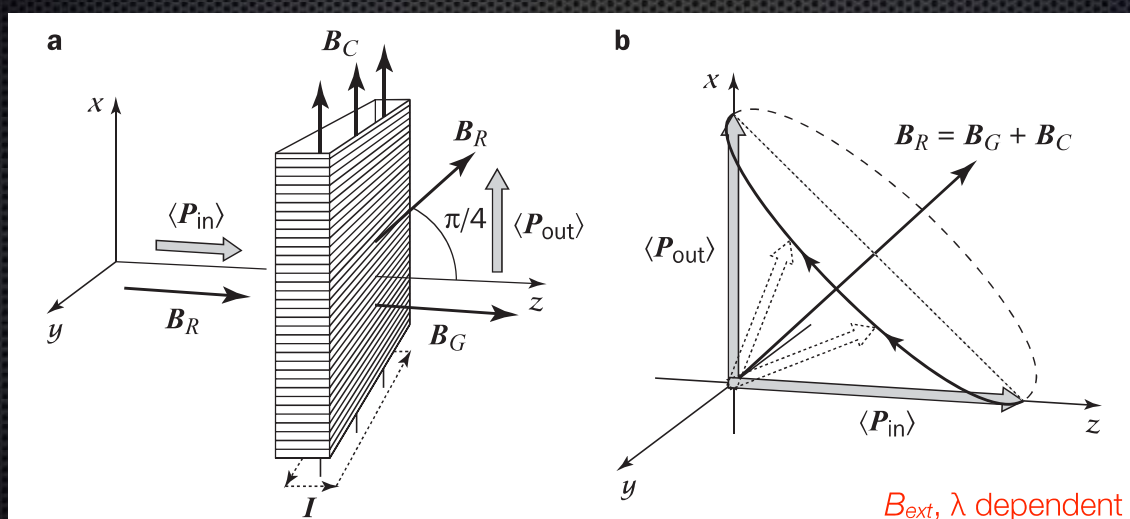
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<http://www.ill.eu>

Spin flippers (Mezei)

- Example of a $\pi/2$ flipper: the neutrons enter and exit the coil non-adiabatically.



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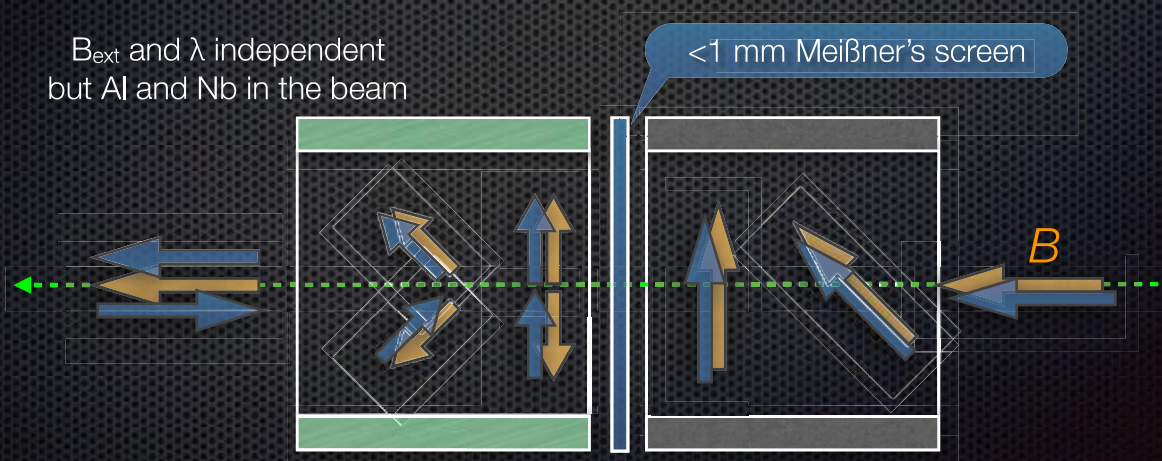
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Spin flippers (Tasset)

- The neutrons enter the second coil non-adiabatically. Perfect flipper even in 400 Gauss stray field.

B_{ext} and λ independent
but Al and Nb in the beam



Spin flippers (Tasset)

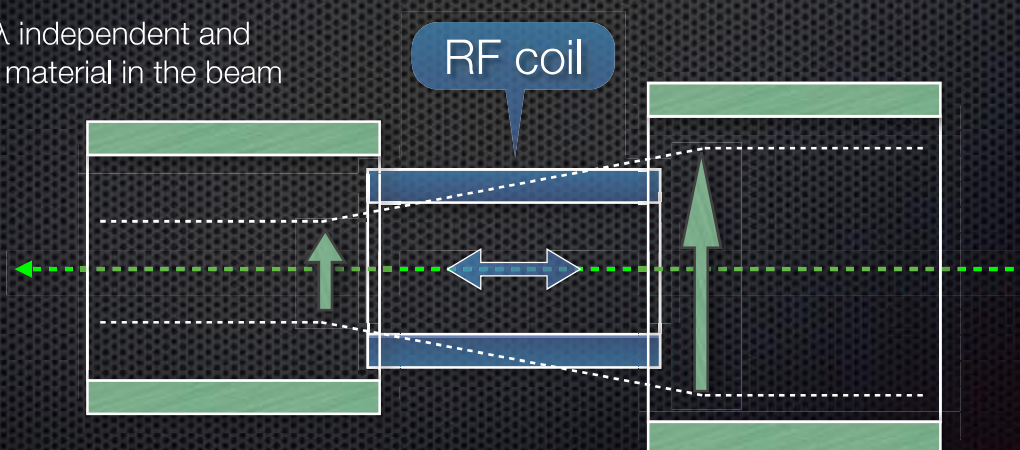
- 99.9% efficient
- $\lambda > 0.4 \text{ \AA}$
- 10L liquid He
- 3 weeks He autonomy
- Al & Nb in beam
- To be cooled down in zero field !



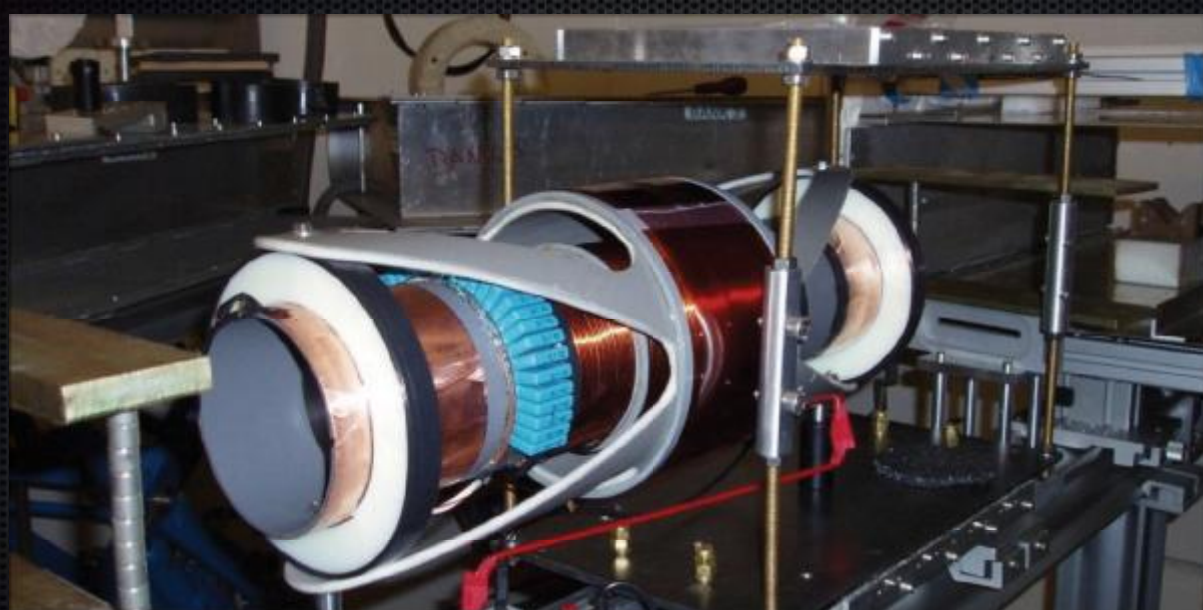
Spin flippers (RF adiabatic)

- In the rotating frame of the neutron, the polarisation follows the effective field and rotates adiabatically.

λ independent and
no material in the beam



Spin flippers (RF adiabatic)



153 kHz/10 A adiabatic flipper for $\lambda > 0.4 \text{ \AA}$

³He spin filters

- Spin filters are characterised by their opacity:

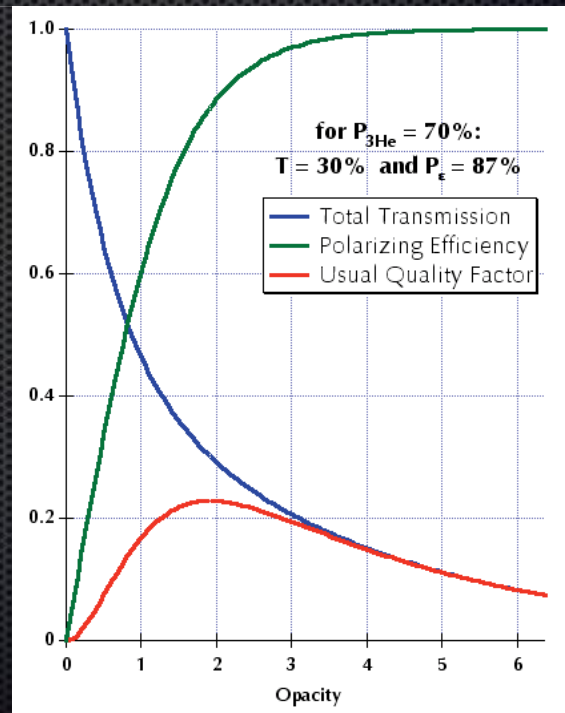
$$\mathcal{O} = N \ell \sigma_{\parallel}$$

$$\simeq 0.0797 p[\text{bar}] \ell[\text{cm}] \lambda[\text{\AA}]$$

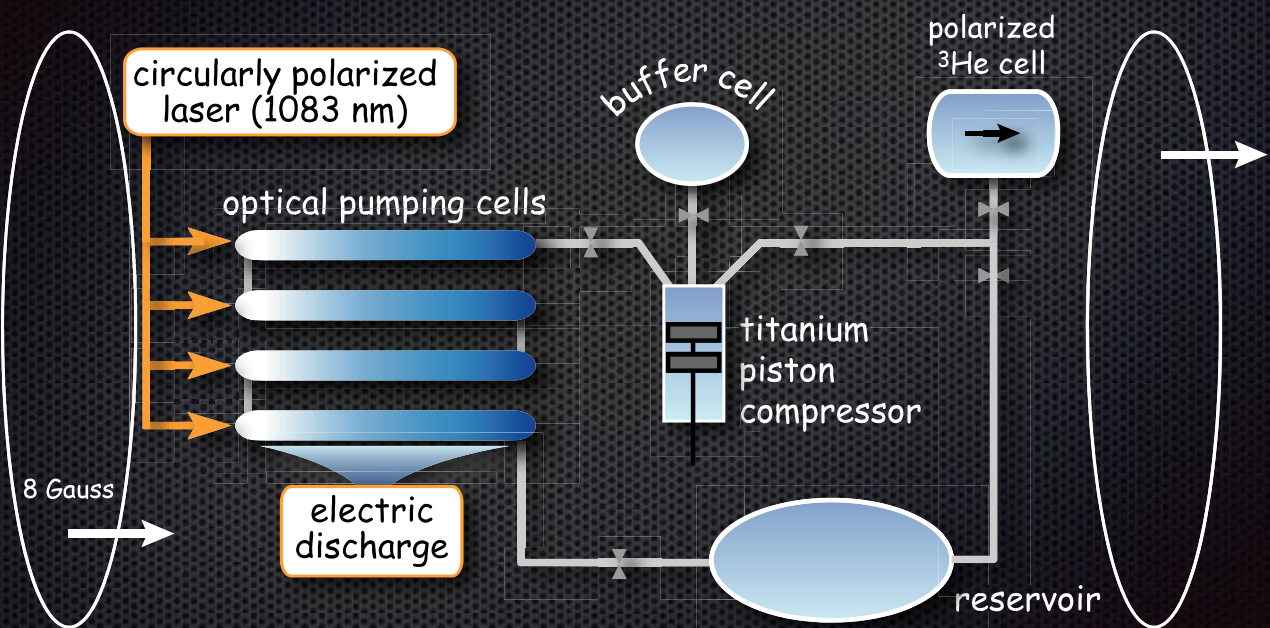
- The total transmission and polarising efficiency are:

$$T_n \propto \cosh(\mathcal{O} P_{3\text{He}})$$

$$P_{\epsilon} = \tanh(\mathcal{O} P_{3\text{He}})$$



³He spin filters



One way to polarise ³He nuclei: metastable exchange optical pumping

^3He spin filters

MEOP station developed at ILL



^3He spin filters

- The main difficulty resides in the ability to build good cells and preserve the ^3He polarisation on the instrument:



³He spin filters

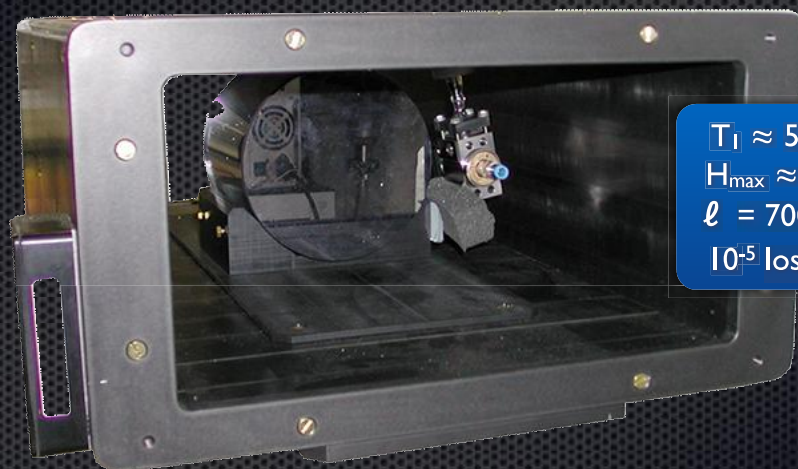
- The main difficulty resides in the ability to build good cells and preserve the ³He polarisation on the instrument:

$$\frac{1}{T_1} = \frac{1}{T_{wall}} + \frac{1}{T_{field}} + \frac{1}{T_{dipolar}}$$
$$= \gamma \frac{S}{V} + \frac{14\,400}{p [\text{bar}]} \left(\frac{1}{B_0} \frac{\partial B_{\perp}}{\partial r_{\perp} [\text{cm}]} \right)^2 + \frac{p [\text{bar}]}{830}$$



i.e. essentially $\frac{1}{B_0} \frac{\partial B_{\perp}}{\partial r_{\perp}} \ll 5 \cdot 10^{-4} \text{ cm}^{-1}$

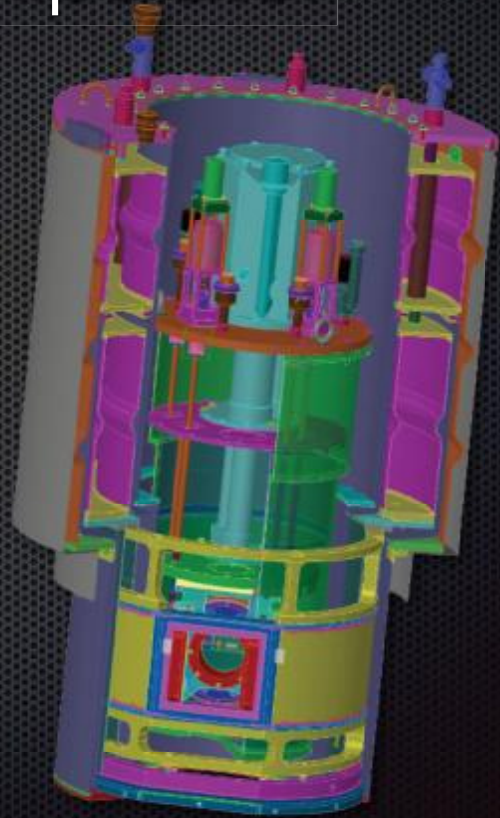
³He spin filters



$T_1 \approx 500 \text{ h}$
 $H_{\text{max}} \approx 10 \text{ G}$
 $\ell = 700 \text{ mm}$
 $10^{-5} \text{ loss/flip}$

Ø140 Si-windowed cell, pneumatic valve,
permanent static field, flipper included.

Polarisation manipulator



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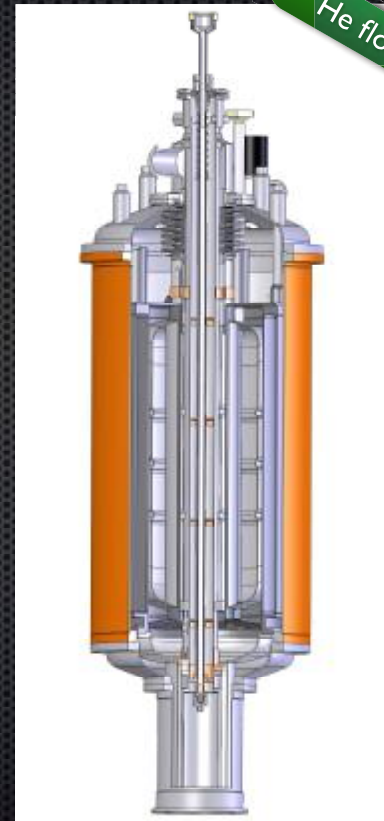
Cryogenics

- **Cryostats**

1.5 / 2.8 to 320 K

- **Cryofurnaces**

1.5 to 550 / 650 K



Cryogenics

- **Cryostats**

1.5 / 2.8 to 320 K

- **Cryofurnaces**

1.5 to 550 / 650 K

- **Dry cryostats**

1.8 to 320 K



Cryogenics

- **Cryostats**

1.5 / 2.8 to 320 K

- **Cryofurnaces**

1.5 to 550 / 650 K

- **Dry cryostats**

1.8 to 320 K with JT

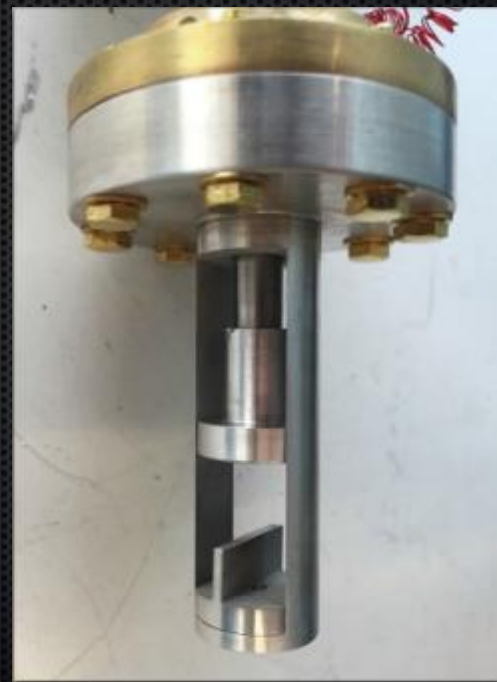
2.7 to 620 K without JT

- **Goniometers, de-twinning**



Cryogenics

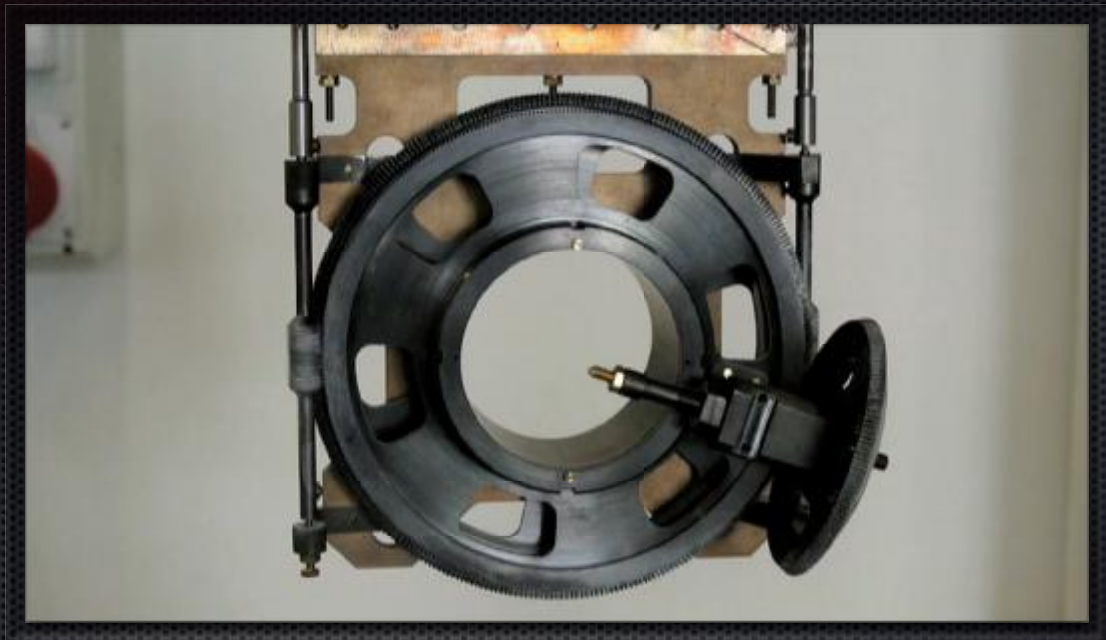
De-twinning crystals remotely inside cryostats





Goniostick

To orient samples in cryostats and cryomagnets



Cryocradle

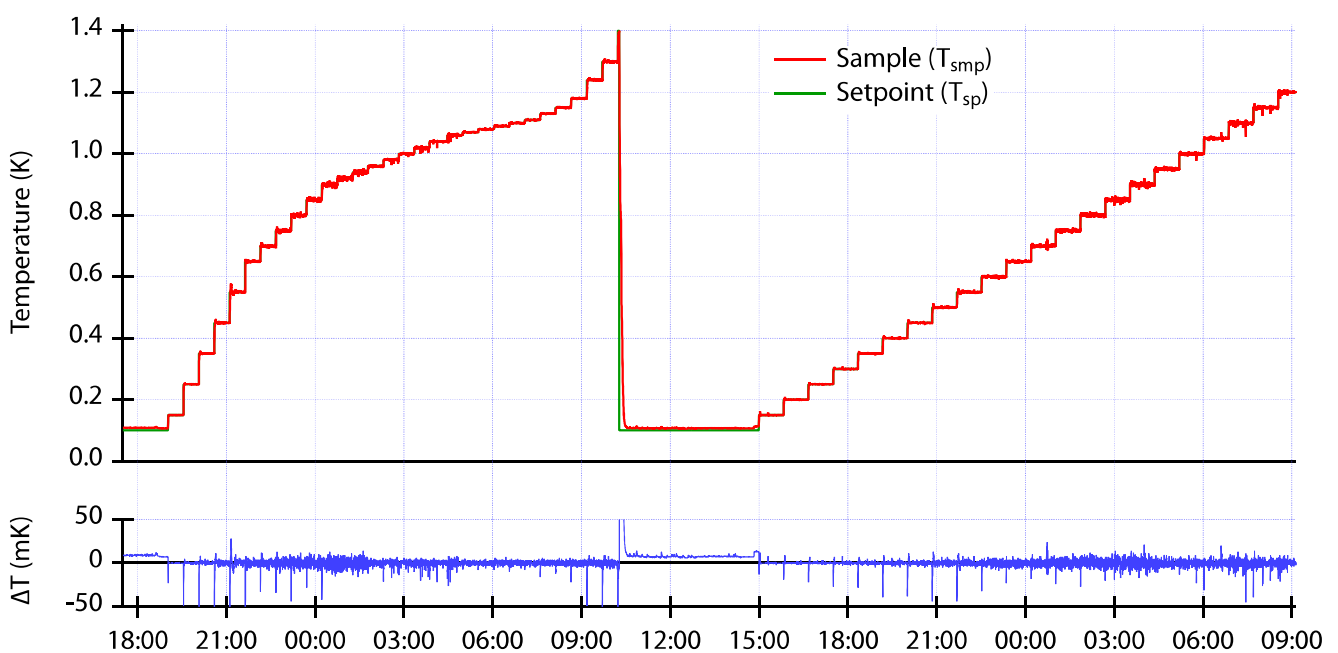
To orient samples in polarimeters down to 3 K

Cryogenics

- **Dilution fridges**
15 / 35 mK to 320 K
- **^3He fridges**
350 mK to 320 K



Cryogenics



Cryogenics

- **Dilution fridges**

15 / 35 mK to 320 K

- **^3He fridges**

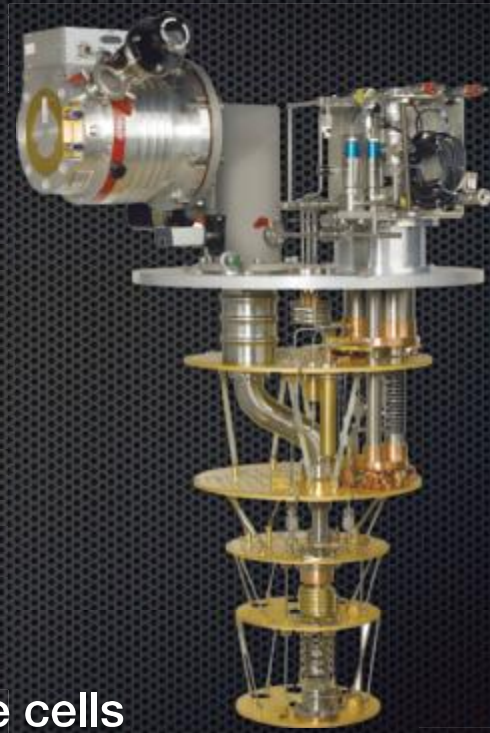
350 mK to 320 K

- **4-circle dilution fridge**

100 mK closed-cycle

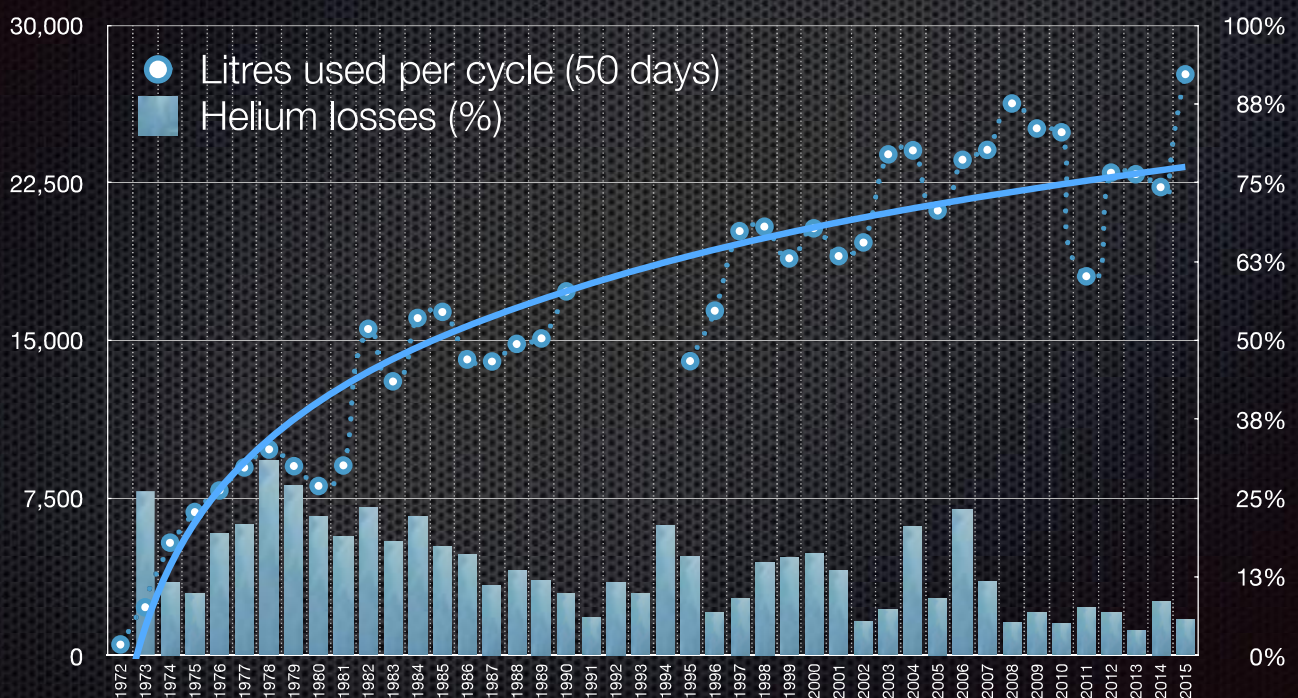
- **For large samples/pressure cells**

Closed-cycle dilution cryostat



Cryogenics

It is worth recovering helium: ≈ 14 M€ saved since 1972



High B fields

- **Hor. field cryomagnets**

 - 17 T static — $T > 100$ mK

 - 40 T pulsed — $T > 2$ K

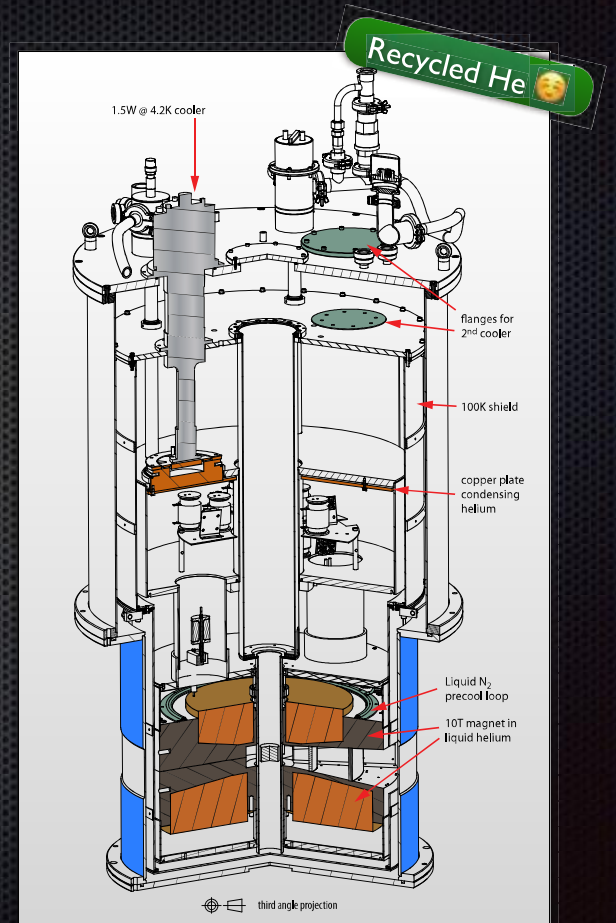
- **Vert. field cryomagnets**

 - 7 T with sapphire windows

 - 11 T with large openings

 - 15 T / 30 mK — 300 K

 - Zero-boil-off option



High B fields

- **Hor. field cryomagnets**

 - 17 T static — $T > 100$ mK

 - 40 T pulsed — $T > 2$ K

- **Vert. field cryomagnets**

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High temperature

- **Standard furnaces**
 - 320 to 1900K
 - V, Nb resistors
 - Sapphire windows
 - mirror furnace
- **Automatic power racks**
- **Furnace for cradles**
- **Laser heating, levitation**



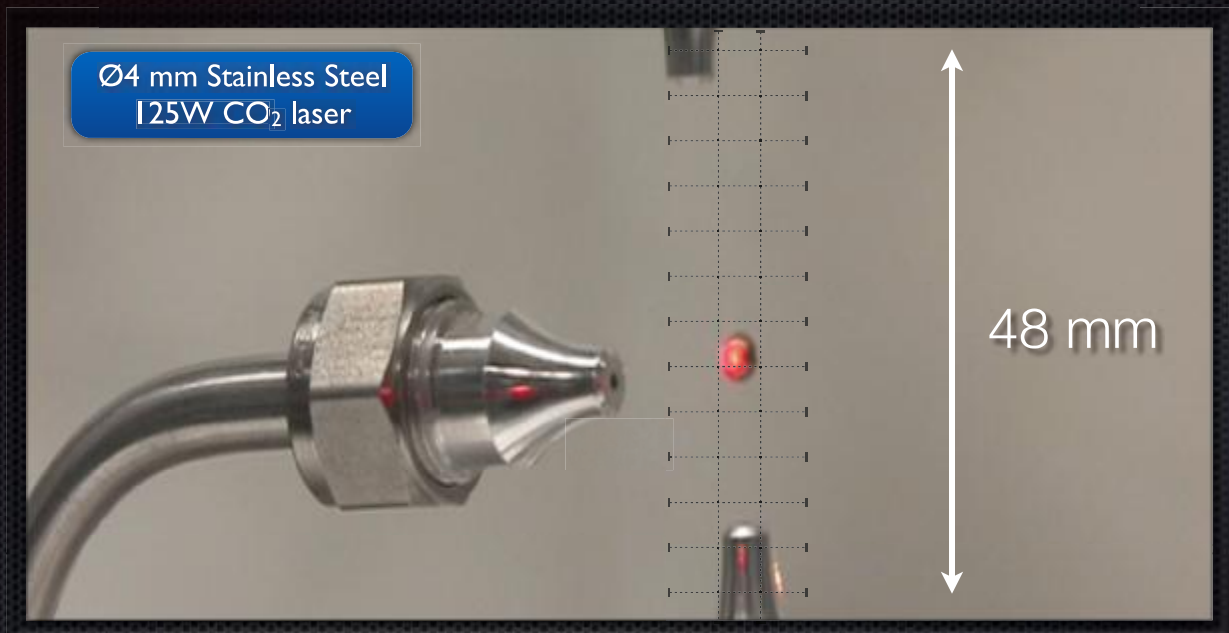
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High temperature

- Standard furnaces
- Automatic power racks
- Furnace for cradles
 - 300 to 1100 K in 1h30
 - 30' to cool down to 650 K
 - 30' to replace the heater
- Laser heating, levitation



Aerodynamic levitation

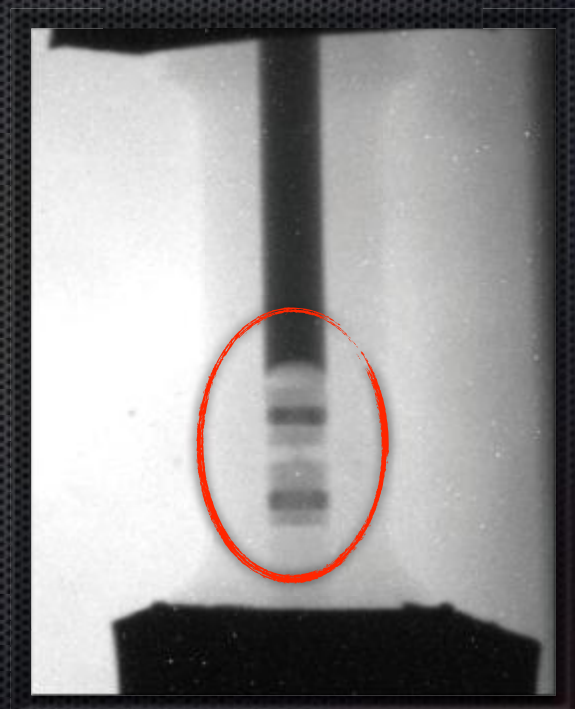
Collaboration with CEMHTI, SIMAP and Soleil

High pressure

- Which materials can we use in the beam ?
 - Aluminium 7049A-T6: transparent but strong signal into detector above $\Delta E \approx 5$ meV
 - Hardened CuBe alloy: less signal above $\Delta E \approx 5$ meV but activates quickly in high flux beams
 - TiZr alloy: no Bragg peak but incoherent scattering
 - Sapphire: transparent but fragile
- Pressure transmitter: He, Fluorinert FC-770...

High pressure

- **Clamps (fluorinert)**
1 to 3 GPa
- **Cells (liq. or gas)**
0.5 to 25 GPa
at 3 K in 7 hours
Automatic control
- **Gas loading capability**



High pressure

Collaboration
with S. Klotz (IMPMC)

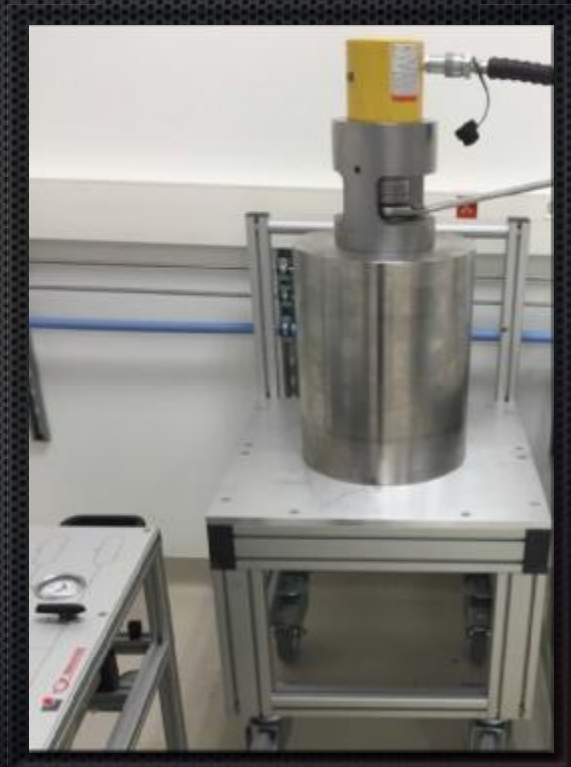
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High pressure

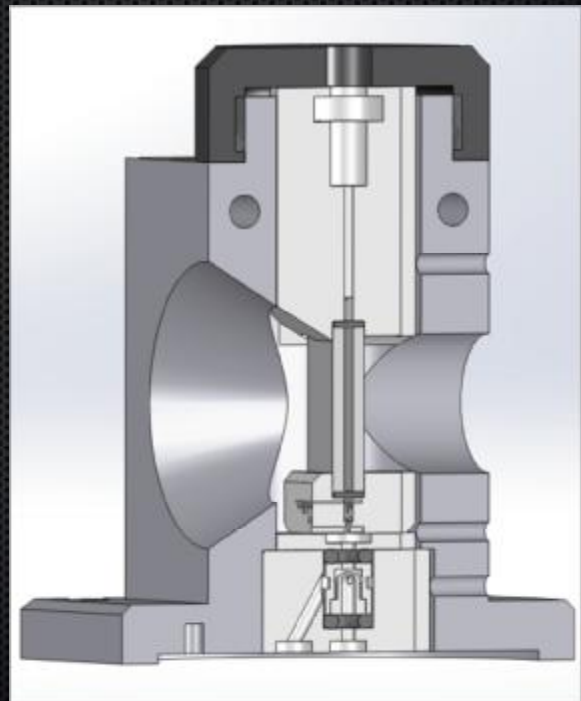
Collaboration
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at 3 K in 7 hours
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What else?

- ✦ Stopped-flow heads
- ✦ Liquid-liquid interface cells
- ✦ Humidity chambers
- ✦ High-pressure cells for bio
- ✦ Rheometers, E-field cells
- ✦ Adsorption troughs, etc.



Sample Environment...

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- ✦ Liquid-liquid interface cells
- ✦ Humidity chambers
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- ✦ What do we measure and need ?
 - ✦ Neutron source and guides
 - ✦ Measuring techniques
 - ✦ Energy selectors and polarisers
 - ✦ Sample environments
 - ✦ Neutrons detectors
 - ✦ Data acquisition system

Neutron Detectors

- We cannot directly detect slow neutrons: they carry too little energy and have no charge.
- We need to use nuclear reactions to convert neutrons into energetic charged particles.
- Then, we can use some of the many types of charged particle detectors

Neutron Detectors

- Ionisation mode: Electrons drift to anode, producing a charge pulse with no gas multiplication. Typically employed in low-efficiency beam-monitor detectors.
- Proportional Mode: If voltage is high enough, electron collisions ionise gas atoms producing even more electrons. Gas amplification increases the collected charge proportional to the initial charge produced.
- Other techniques: CCD cameras, image plates (Laue), scintillation detectors ($n + {}^6\text{Li} \rightarrow {}^4\text{He} + {}^3\text{H} + 4.79 \text{ MeV}$).

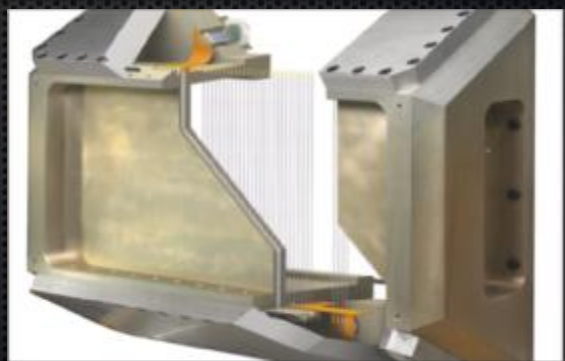
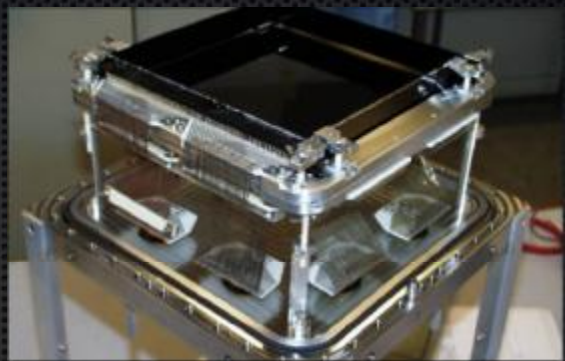
Neutron Detectors

- Spatial resolution is “generally” not an issue, in the range of 1 mm to 1 cm i.e. roughly the sample size
- Fast neutrons, electronics and gammas lead to background noise. Counting mode is more appropriate than integrating mode.
- High detection efficiency required for scattered neutrons, but low efficiency is enough for incident beam.



ToF: 30 m² low-res, low count rate

19x19 cm² high res, high count rate

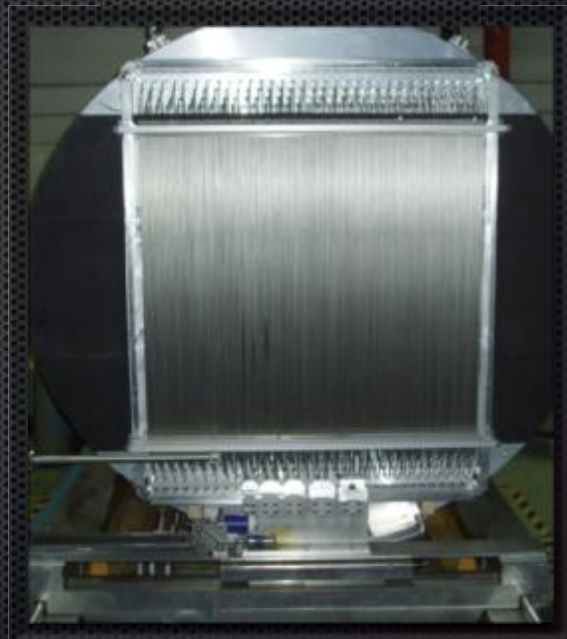


Reflect., SANS: Monobloc multitube

Neutron Detectors



Old XY counter — 200 kHz max.



New 128 PSD counter — x50 better



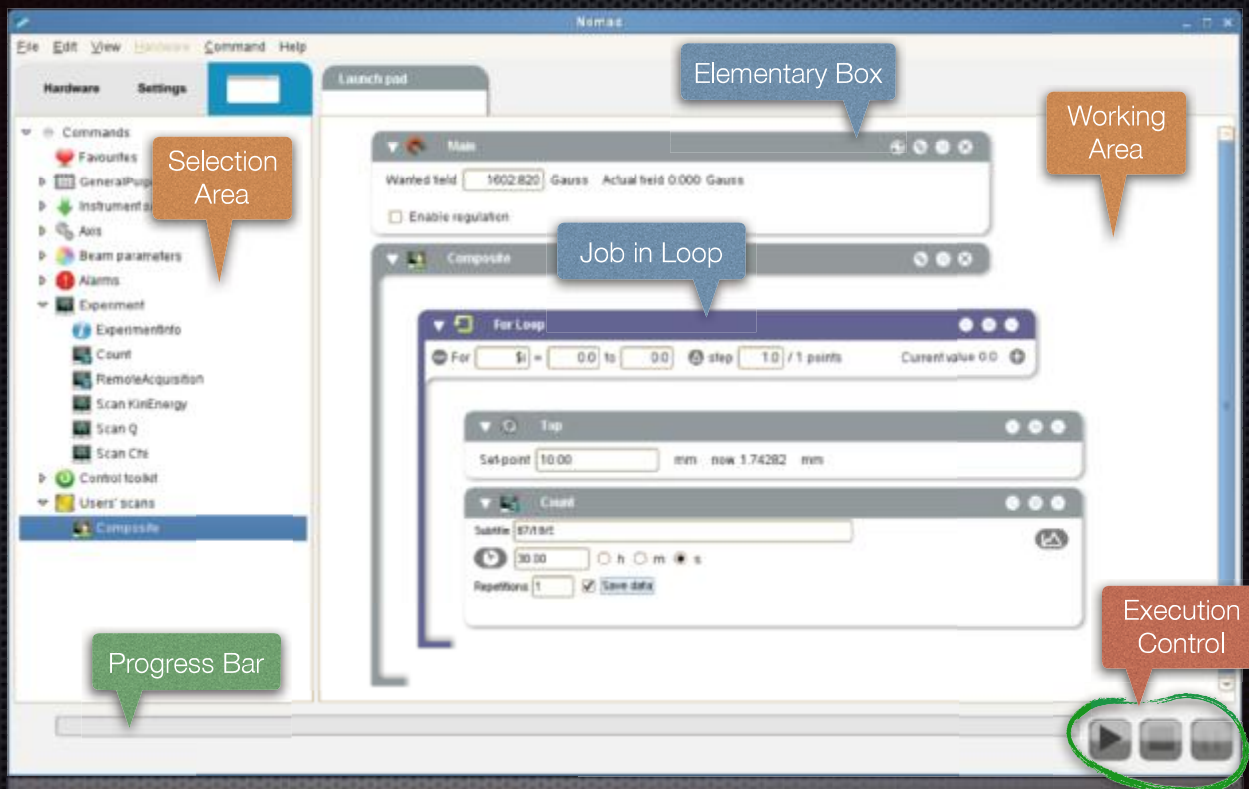
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 - ❖ Neutrons detectors
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Data acquisition system

- ❖ Speaks in physical units, ranges with physical entities
- ❖ Acts as a super-calculator for the local contact to access complex instrument's configurations
- ❖ Provides performance optimiser for fine adjustments or advanced regulations
- ❖ Checks jobs, estimates run-time, execute jobs safely
- ❖ Also provides command-line tools, remote access, etc.

Data acquisition hardware

- ✦ VME crates (low power)
- ✦ NIM crates (high power)
- ✦ Power supplies for DC and stepper motors, flippers, guiding fields, etc.
- ✦ Sample env. controllers



Special thanks to...

I. Anderson — ORNL (USA)
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B. Guérard — Neutron Detectors, ILL
M. Kreuz — Neutron Guides, ILL
P. Mutti — Instrument control, ILL

Thank you for your attention

