

# Sample environment for inelastic neutron scattering

Jiří Kulda

Institut Laue-Langevin, Grenoble, France



#### Sample environment



#### Temperature:

dilution insert > 15 mK
 Orange cryostat 1.5 - 300 K
 cryofurnace 2 - 500 K
 resistive heating 300 - 1400 K
 optical (mirror) 800 - 2000 K

#### **Pressure:**

 $\begin{array}{lll} \bullet & \textit{continuously loaded} & \textit{p < 3-5 kbar} \\ \bullet & \textit{clamp} & \textit{p < 30 kbar} \\ \bullet & \textit{uniaxial} & \textit{F < 10000 N} \end{array}$ 

ALL works down to  $T \approx 2 \text{ K!}$ 

• Paris-Edinburgh cell p < 100 kbar T > 4 K

#### Magnetic field:

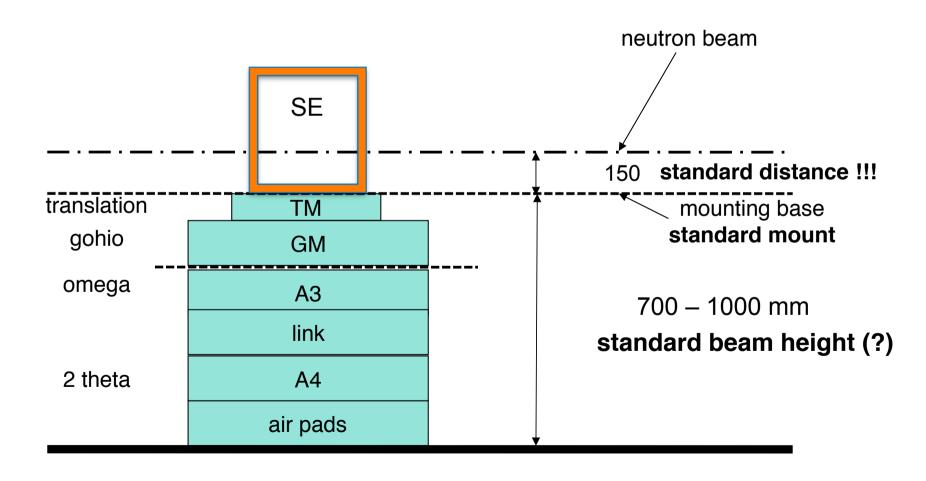
• CRYOPAD  $\approx 15 \text{ mG}$ • cryomagnets 1 - 15 T • pulsed field < 40 T

+ electric field, gas sorption, ....



#### Sample environment

#### **Standard sample mounting system**



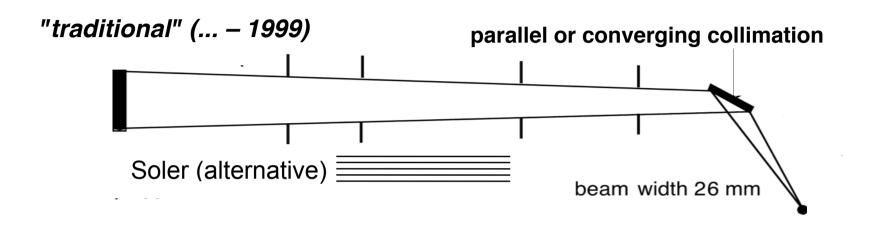


## Layout of the talk

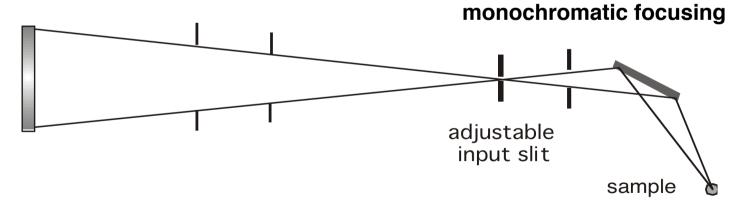
- 1. Optics
- 2. High pressures
- 3. Magnetic fields
  - 1. Zero field (CRYOPAD, MUPAD, ...)
  - 2. Conventional cryomagnets
  - 3. Ultra-high fields
- 4. Conclusions



## TAS layout



#### "modern" (2000 - ...)



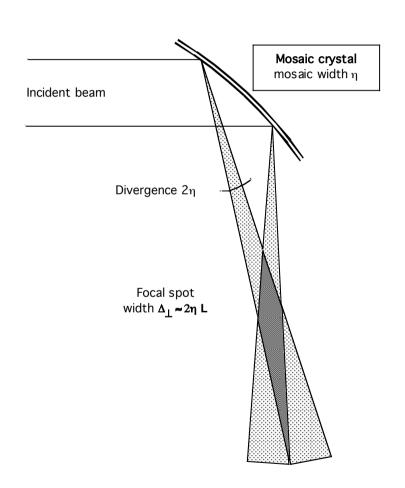
intensity gain ≈ 30x

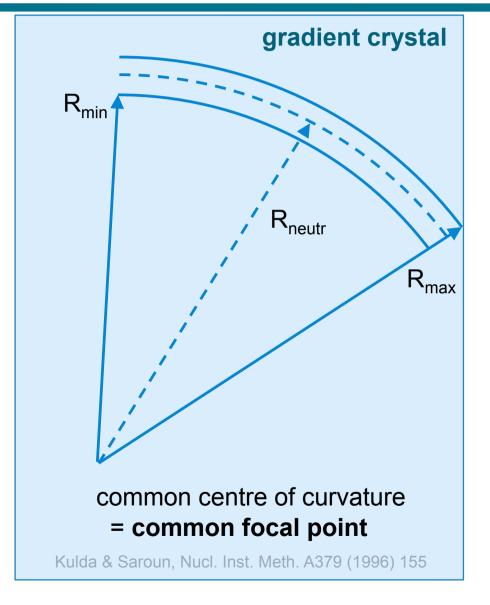
sample size > 1 cm3 → 100 mm3



## Focusing properties

#### mosaic crystal



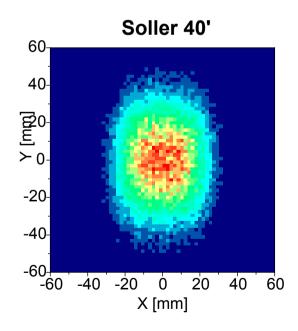




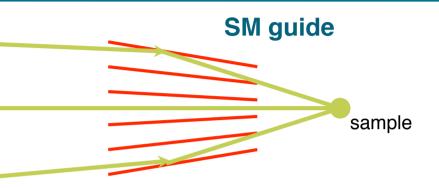
### SM microfocusing

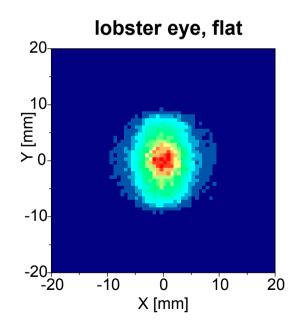
#### 2D lobster eye optics:

- PG 002, k=1.55A-1
- supermirror *m*=3
- 11 x 15 slits (HxV)
- length = 300 mm
- sample distance = 300 mm



 $\Phi = 0.9 \cdot 10^6 / \text{mm}^2$ 

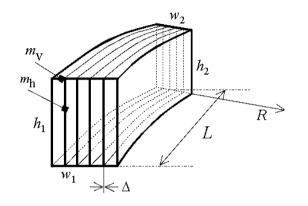




 $\Phi = 9.5 \cdot 10^6 / \text{mm}^2$ 

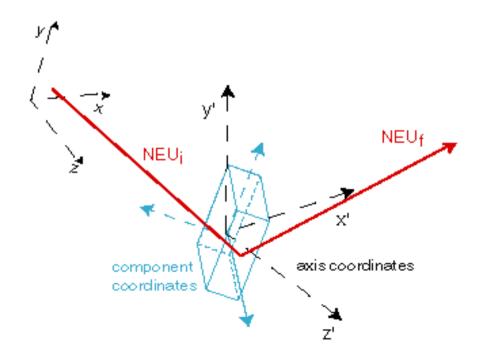


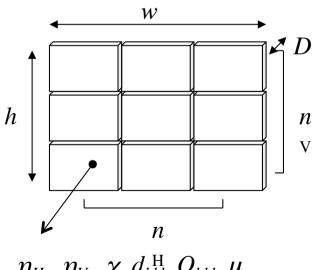
#### RESTRAX



- neutron ray-tracing or multi-Gaussian convolution
- diffraction/reflection optics of neutron instruments
- realistic crystal description (mosaic, elastically bent)
- highly optimized F77/F95 code

http://omega.ujf.cas.cz/restrax

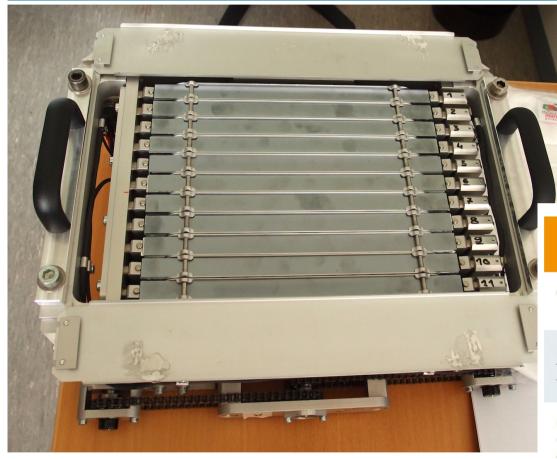




$$\eta_H$$
,  $\eta_V$ ,  $\chi$ ,  $d_{hkl}^H$ ,  $Q_{hkl}$ ,  $\mu$ , ...



## Si (111) vers. PG(002)



#### IN1, IN8, IN20, ThALES

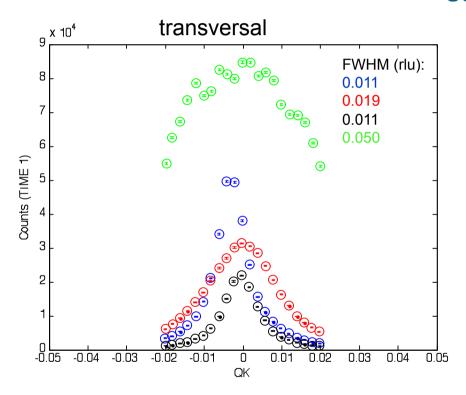
Monochromator				
Crystal	W x H (mm <sup>2</sup> )	ki/Å <sup>-1</sup>	flux/10 <sup>8</sup> n cm <sup>-2</sup> s <sup>-</sup>	
PG (002) double focusing, three faces	233x197	2.662 4.1	2.0 6.5	
Cu (200) variable double-focusing, anisotropic mosaic (h:25', v:10')	233x197	4.1 7.0	4.6 3.0	
Si (111) bent perfect crystals, fixed horiz. curvature optimized for k=3.5Å <sup>-1</sup>	180x197	2.662 4.1	0.8 3.4	

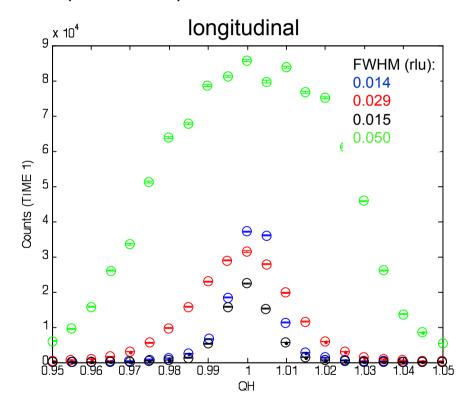
monochromatic flux  $\approx 1/3$  PG



### Si (111) vers. PG(002)

#### **Bragg width (PMN 100)**

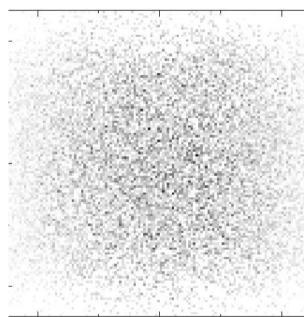




PG-PG open DTR 40
PG-PG 40' – 40' DTR 40
Si-Si open DTR 10
Si-Si 40' – 40' DTR 40



## Microfocusing crystal optics



PG002 horizontal focus (RESTRAX ray-tracing)



Paris-Edinburgh High pressure cell



Si111 horizontal focus (RESTRAX ray-tracing)



### Layout of the talk

- 1. Optics
- 2. High pressures
- 3. Magnetic fields
  - 1. Zero field (CRYOPAD, MUPAD, ...)
  - 2. Conventional cryomagnets
  - 3. Ultra-high fields
- 4. Conclusions



### High pressures

## Paris-Edinburgh cell ILL version

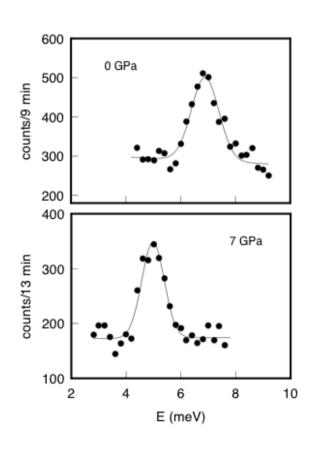
- pressure < 8 GPa
- temperatures > 4 K
- sample volume < 40 mm<sup>3</sup>

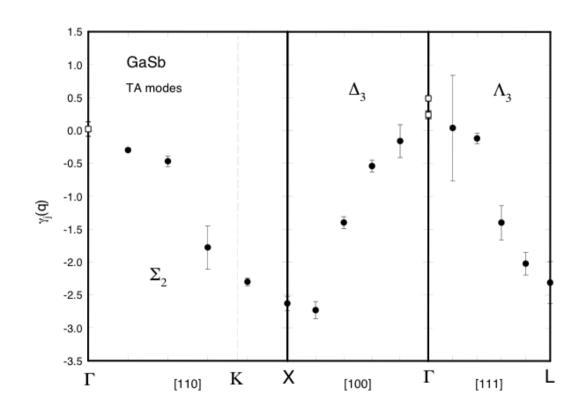




#### GaSb negative Grueneisen parameter

## X-point TA phonon in GaSb at 0 and 7 GPa





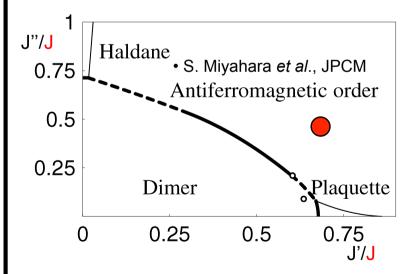
S.Klotz, M.Braden, J. Kulda, P.Pavone, B.Steininger phys. stat. sol. (b) 223 (2001) 441



#### High-pressure Studies of Quantum Magnets

#### Shastry-Sutherland compound SrCu<sub>2</sub>(BO<sub>3</sub>)<sub>2</sub>

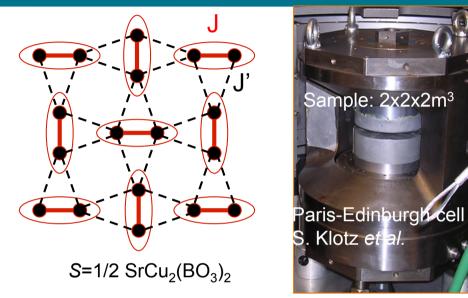
- pressure-control of exchange interactions
- INS measurements of elementary excitations
- magnetic and structural phase transitions

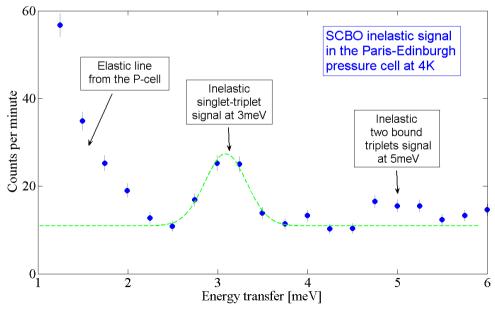


M. Zayed, H.M. Rønnow, EPF Lausanne Ch. Rüegg, UCL

S. Klotz, G. Hamel, IMPMC Paris

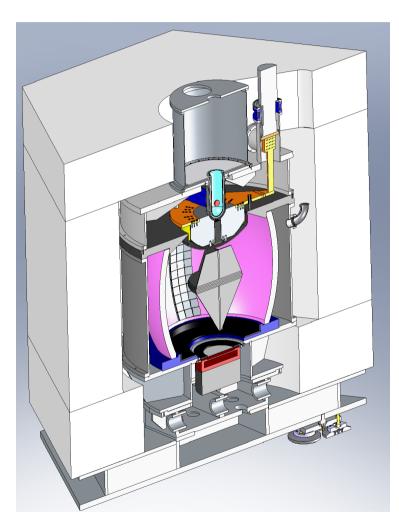
K. Conder, Th. Strässle, PSI







#### Chemical spectroscopy



ILL/Spain co-funding

A. Ivanov et al., ILL 2009-2011

#### **IN1 LAGRANGE**

Be-filter/PG-analyzer  $\Delta E < 1000 \text{ meV}$ 

	BeF	Lagrange
solid angle [sr]	0.06	2.5*
∆E [meV]	3	0.75
transmission	0.7	0.5
background	1	1/30 – 1/10

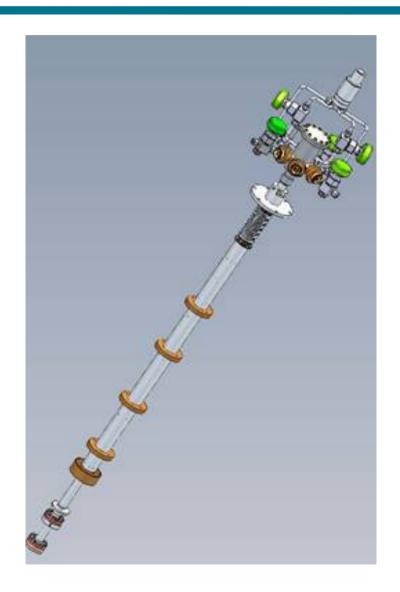
\*) IN5 ≈ 1.8 sr

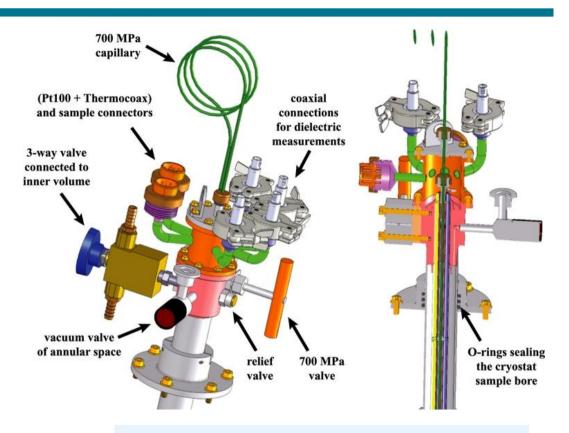
Samples down to 10 μg H 10 mg C

 $^{1}$ H :  $\sigma_{inc}$  ≈ 80 barn



### Gas handling





#### gas sorption Lagrange

- pressure 80 (< 700) bar
- temperatures 1.5 550 K
- sample space ø 49 mm & 70 mm



### Layout of the talk

- 1. Optics
- 2. High pressures
- 3. Magnetic fields
  - 1. Zero field (CRYOPAD, MUPAD, ...)
  - 2. Conventional cryomagnets
  - 3. Ultra-high fields
- 4. Conclusions

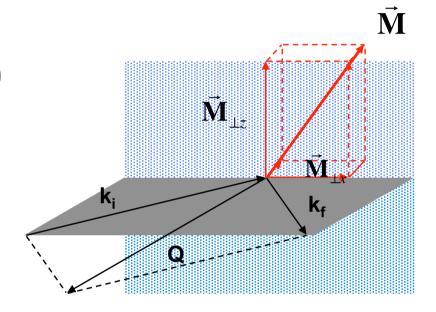


## Magnetic scattering

 $\vec{M}(\vec{Q}) = \sum_{j} \vec{M}_{j}(Q) \exp(i\vec{Q}\vec{r}_{j}) \exp(-W_{j})$ 

• only projection of **M⊥Q** contributes

$$\vec{M}_{\perp} \left( \vec{Q} \right) = \vec{e}_{Q} \times \vec{M} \left( \vec{Q} \right) \times \vec{e}_{Q}$$



"Moon's golden rule":

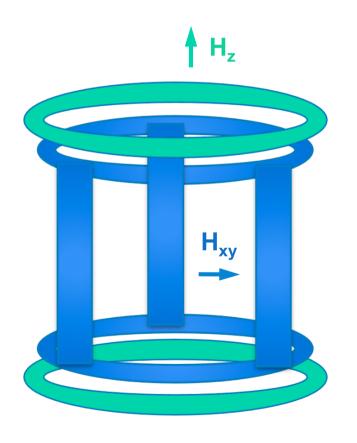
$$\vec{M}_{\perp} \parallel \vec{\sigma}_n$$
 non spin-flip (NSF) U<sup>++</sup>, U<sup>-</sup>

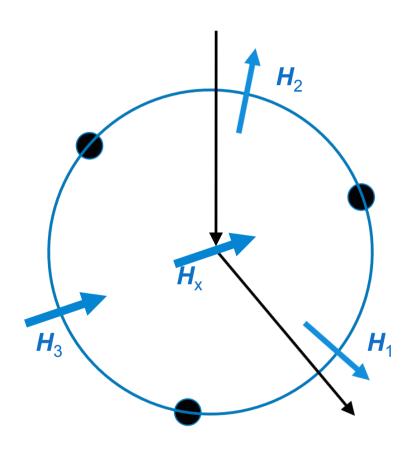
$$\vec{M}_{\perp} \perp \vec{\sigma}_n$$
 spin-flip (SF) U+-, U-+



## 3D diagonal polarisation analysis

#### Triple-dutch & Helmholtz coils



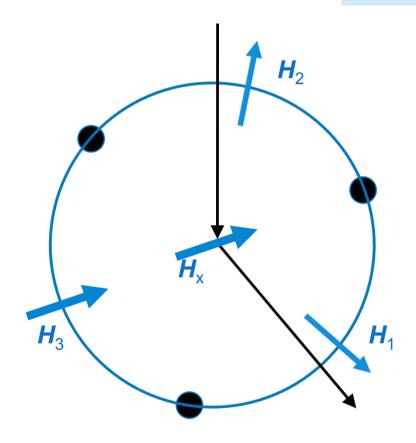


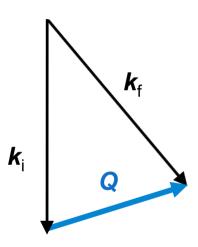
compose guiding field in any direction in 3D



## 3D diagonal polarisation analysis

#### Elastic scattering

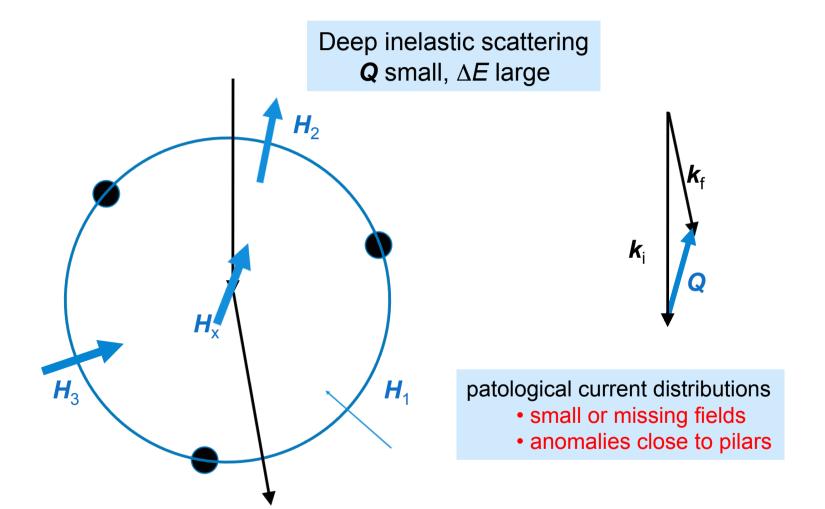




easy current distribution optimisation



### 3D diagonal polarisation analysis

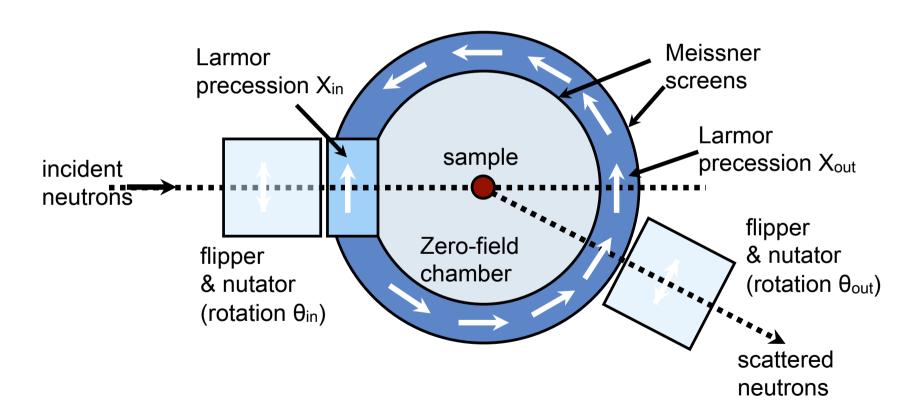


>> use zero-field environment



#### **CRYOPAD** scheme

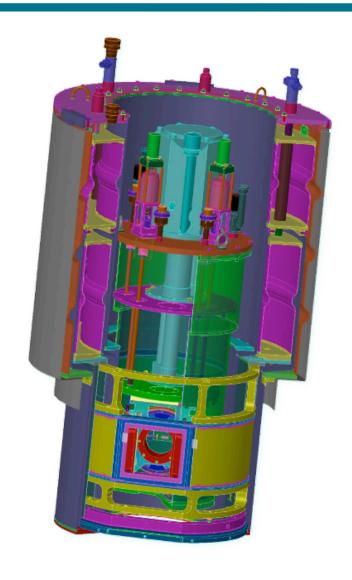
#### CRYOPAD II



F. Tasset et al., Physica B 267}268 (1999) 69



## CRYOPAD design







### Layout of the talk

- 1. Optics
- 2. High pressures
- 3. Magnetic fields
  - 1. Zero field (CRYOPAD, MUPAD, ...)
  - 2. Conventional cryomagnets
  - 3. Ultra-high fields
- 4. Conclusions



## Specifications

- 10T for reasonable cost, reliability
- dilution fridge insert (obviously)
- ease of use

zero-boil-off: we save 25k€ LHe per year, simplify the use cost doubles? NOT! +20%
Reliability? reservoir system reasonable (moving, quenching ..)
BUT: prototype! time scale? other risks? life time?

- with or without self-shielded option

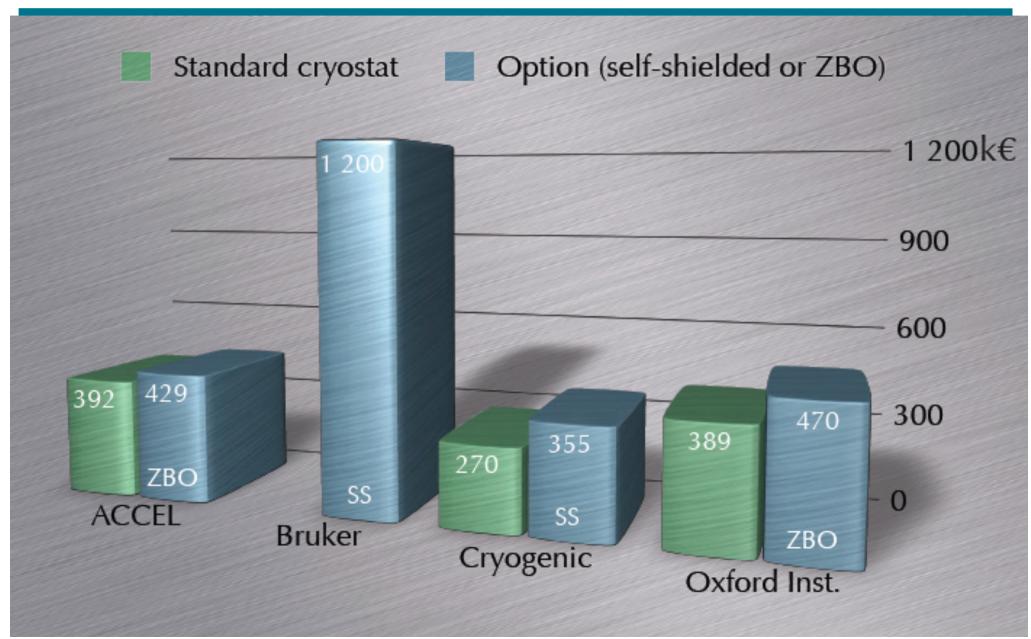
  stray field is reduced by a factor ≈50

  BUT the cost doubles, diameter & weight increases

  WAIT FOR FUTURE
- asymmetric field for polarised neutron beams
- large samples (10mm width, 30mm height)
- large horizontal access: Al rings vers. pillars

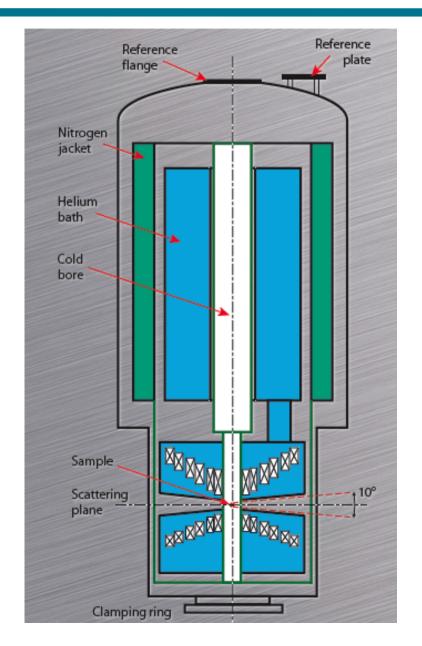


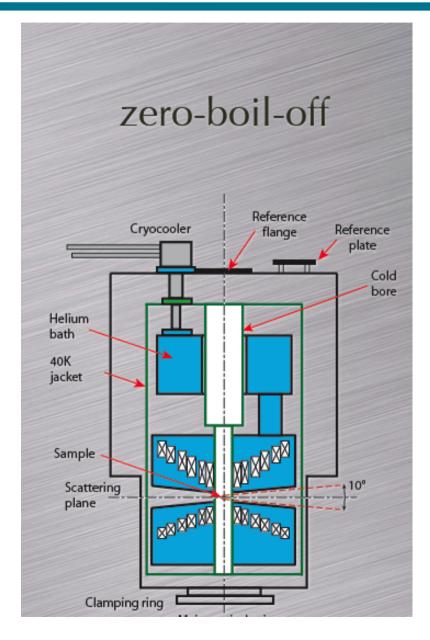
## Price tags

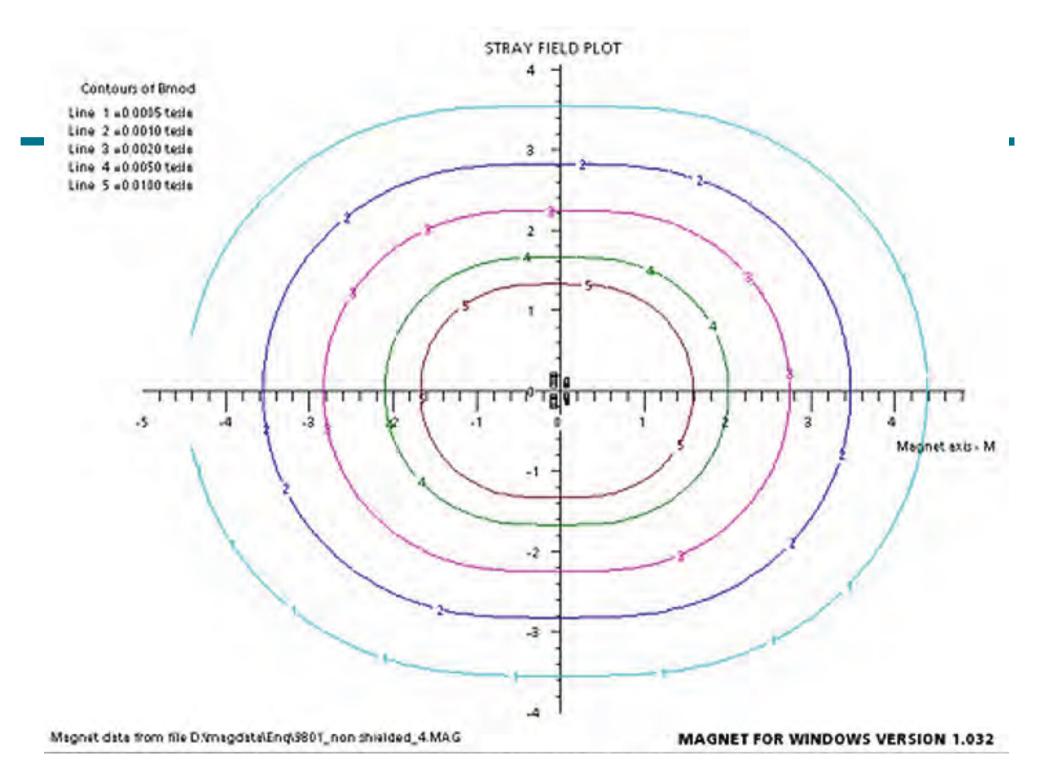


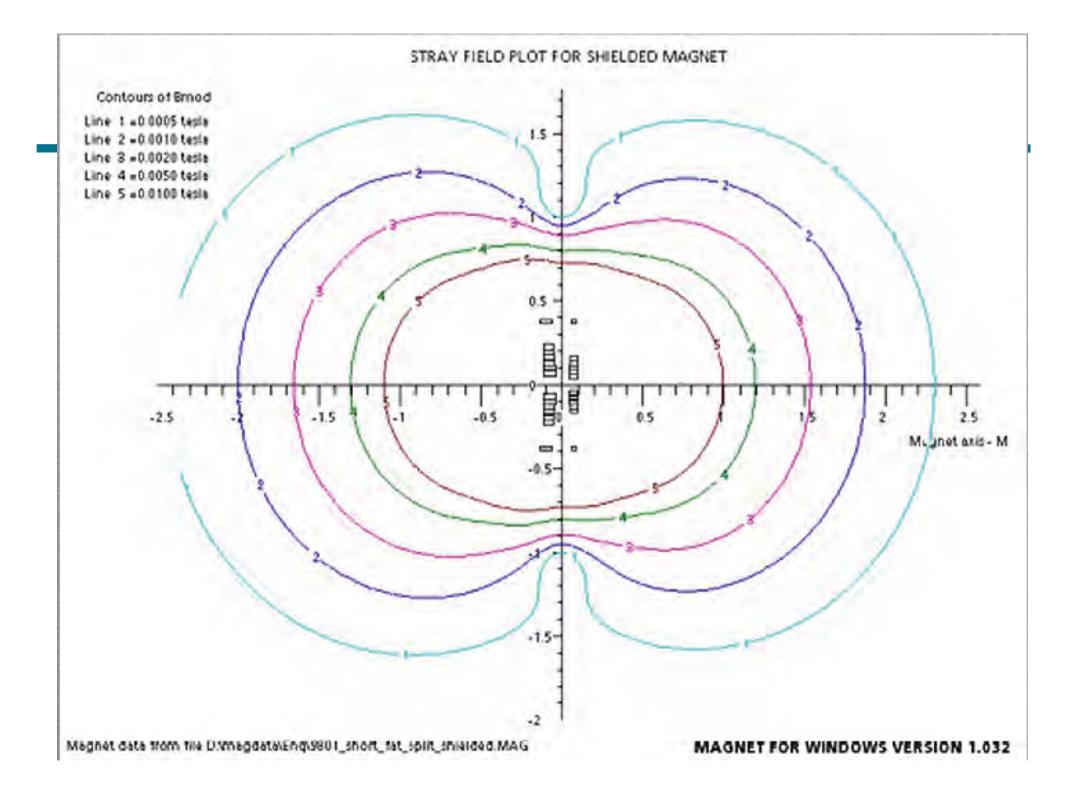


## Cryogenics



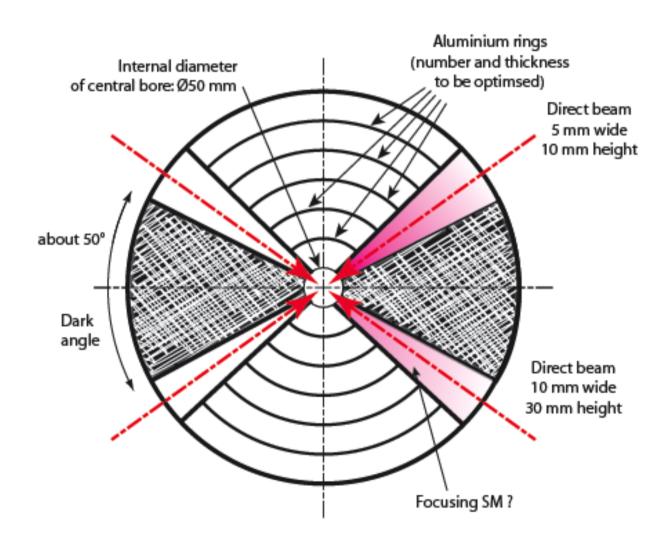








## Beam layout



Here is a design with two pillars [\*]. In this case, one could reduce the thickness of the Al rings. The question is to determine how much. I guess that to a first approximation it is proportional to the relative proportion of angle covered. The gain might be of the order of 20%–30% maximum.

The problem I see is the implementation of the supermirrors. As they would be installed inside the cryomagnet, they cannot be removed between experiments or cycles. They would be in the direct beam, leading to a production of background. On the other hand, they might help you increase the flux when using small samples.

[\*] On this sketch, the pillars are not well drawn and might cut the incident beam. This sketch is for helping discussions only



## 10T recondensation magnet





## Layout of the talk

- 1. Optics
- 2. High pressures
- 3. Magnetic fields
  - 1. Zero field (CRYOPAD, MUPAD, ...)
  - 2. Conventional cryomagnets
  - 3. Ultra-high fields
- 4. Conclusions

### **Ultra-high** magnetic fields > 20 T

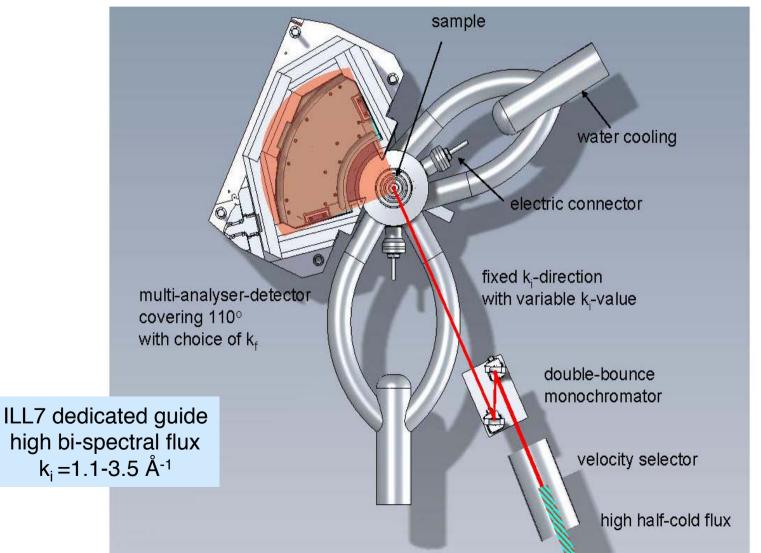
#### Scientific case

- normal state of high-Tc superconductors
- quantum limit of certain metals
- nature of hidden order in URu<sub>2</sub>Si<sub>2</sub>
- static and dynamic correlations in true Bose-Einstein condensates
- spin supersolidity
- nature of correlations at and in between the plateau phases of the two-dimensional Shastry-Sutherland lattice SrCu<sub>2</sub>(BO<sub>3</sub>)<sub>2</sub>
- static and dynamic correlations in quadrupolar spin-nematic Luttinger liquids
- origin of electric polarisation changes at 20-30 T in certain multiferroics
- evolution of magnetic order near charge order melting in Na<sub>0.5</sub>CoO<sub>2</sub> near 30 T.



### HIFI - HIgh-FIeld Diffraction/Spectroscopy

#### **ILL/ESRF** high-field installation



*B* ≈ 30-35 T

 $P_{el} \approx 40 \text{ MW}$ 

power plant cost ≈ 10 - 15 M€

> electricity cost ≈ 10 M€/yr



#### **UHF** magnet Oxford Instruments

## New era in high field superconducting magnets – opening new frontiers in science, nanotechnology and materials discovery 08 January 2018

Oxford Instruments is delighted to congratulate our partners at the National High Magnetic Field Laboratory (NHMFL) on the successful demonstration of a 32 Tesla all-superconducting user magnet on 8th of December 2017. Achieving this major milestone is a step change in high field, compact magnets and important for research into new science and materials discovery. It enhances our understanding and knowledge of superconducting and nanomaterials, leading to new nano-devices and applications.

The 32 T superconducting system is made from two primary sections, an outsert section delivering 15 Tesla in a 250 mm wide bore magnet developed by Oxford Instruments Nanoscience (OINS) using advanced Low Temperature Superconductor (LTS) materials operating at 4.2 Kelvin and an insert section delivering 17 Tesla in 34 mm cold bore developed by our colleagues at NHMFL using advanced High Temperature Superconductor (HTS) materials manufactured by Superpower Inc. Both sections were integrated by NHMFL team in a cryogenic system developed by OINS.

#### 32 T solenoid $>> \approx 22 - 25$ T split coil (?)

key enabling technology: Bi-2212 round wire

Bi-2212 = Bi<sub>2</sub>Sr<sub>2</sub>CaCu<sub>2</sub>O<sub>8+x</sub>



size, weight & operation costs
≈ "normal" self-shielded cryomagnet





### Layout of the talk

- 1. Optics
- 2. High pressures
- 3. Magnetic fields
  - 1. Zero field (CRYOPAD, MUPAD, ...)
  - 2. Conventional cryomagnets
  - 3. Ultra-high fields
- 4. Conclusions

#### Recommendations

#### **Strategic decisions:**

- unification of mechanical interfaces
- adapted beam optics
- cryogenic policy: wet vers. dry
- non-magnetic environment

& progressive procurement policy



See you in



6

Events

Days

DAYS

148

15

**Keynote Speakers** 

Register