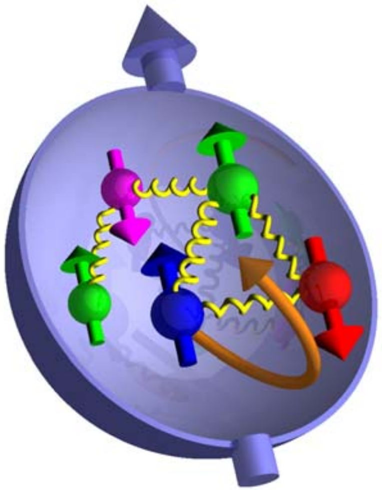
Neutron Spin Precession in Samples of Polarised Nuclei
and
Neutron Spin Phase Imaging

Florian Piegsa

Institut Laue-Langevin, June 9th 2009



- Fundamental neutron physics with polarised nuclei:

  The nd-Experiment

- A "spin-off" project:

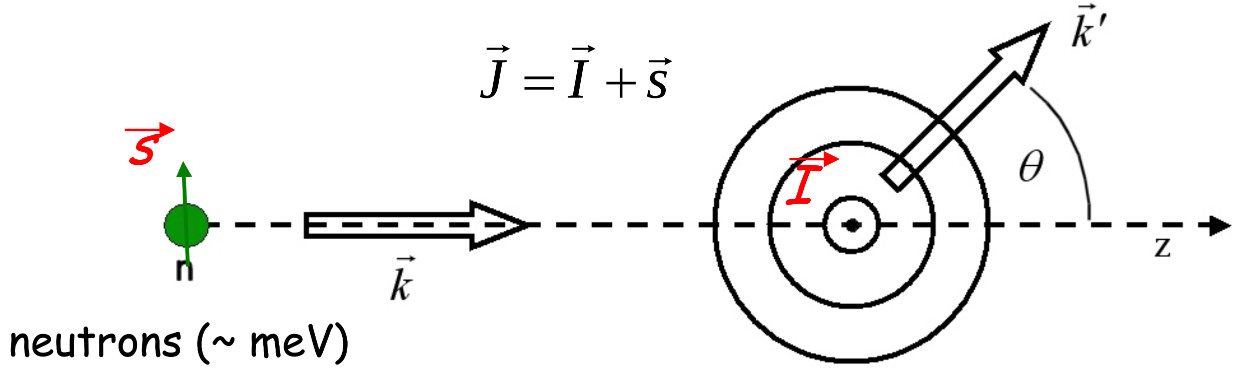
  Neutron Spin Phase Imaging

The nd-Experiment

a high-accuracy measurement of the spin-dependent nd-scattering length

B. van den Brandt, H. Glättli, P. Hautle, J. Kohlbrecher,
J.A. Konter, F.M. Piegsa and O. Zimmer

neutron scattering length



$$b = b_c + \frac{2\, b_i}{\sqrt{I(I+1)}}\, \vec{s}\cdot\vec{I}$$

b_c = coherent / spin-independent scatt. length
b_i = incoherent / spin-dependent scatt. length

Interesting for **Effective Field Theories**:
(3 nucleon system - Neutron + Deuteron)

$$b_{2,d} = b_{c,d} - \sqrt{2}\, b_{i,d}$$

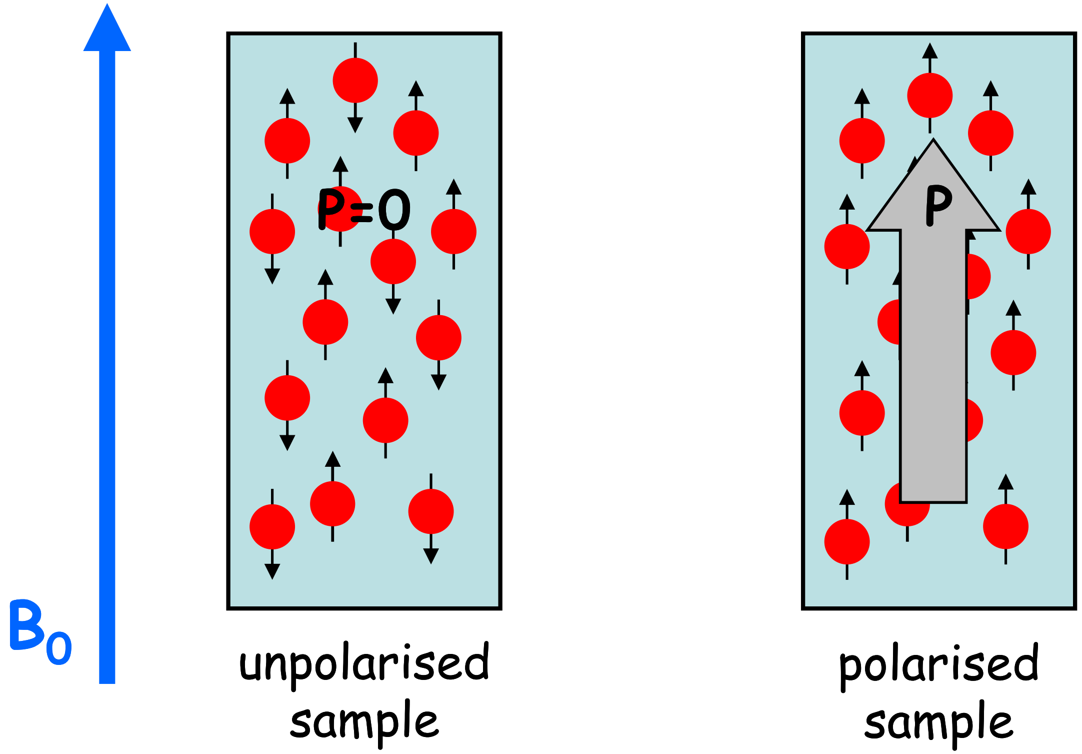
Present knowledge: $b_{i,d}$ = (4.033 ± 0.032) fm ⇨ $b_{2,d}$ = (0.96 ± 0.05) fm

Ultimate Goal: improve accuracy of $b_{i,d}$ ⇨ $b_{2,d}$ ~ 1%

[Dilg et al., PLB **36** (1971) 208]
[Schoen et al., PRC **67** (2003) 044005]

pseudomagnetic precession of the neutron spin

spin-dependent strong interaction



[V. Barychevsky, M. Podgoretsky, JETP **20** (1965) 704]
[A. Abragam et al., PRL **31** (1973) 776]

pseudomagnetic precession of the neutron spin

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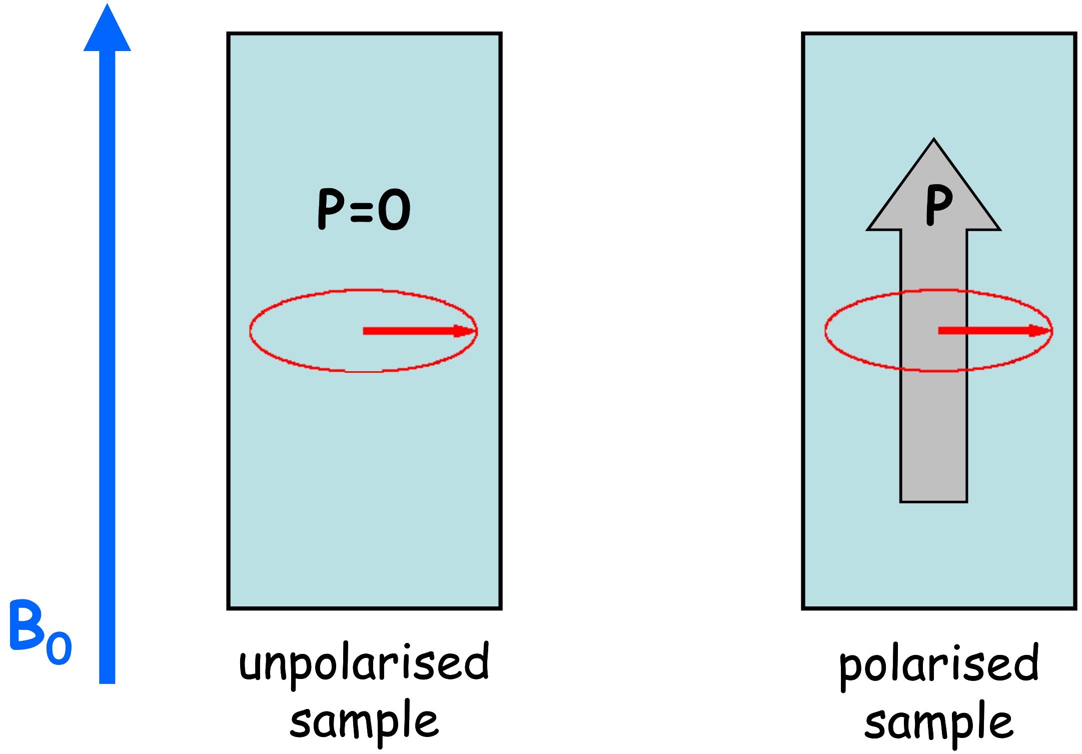


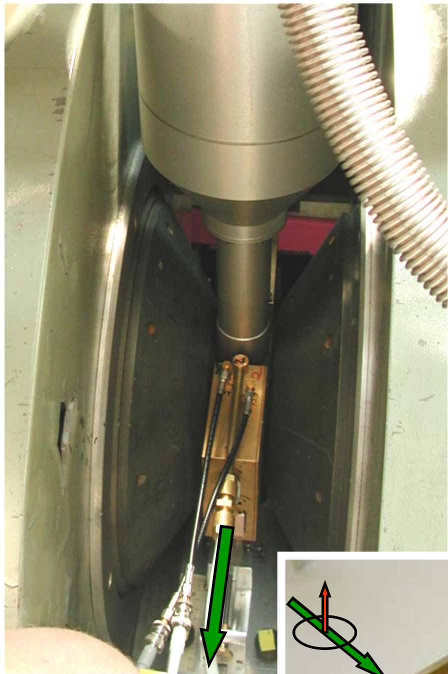
$\omega_{Larmor} = \omega_0$

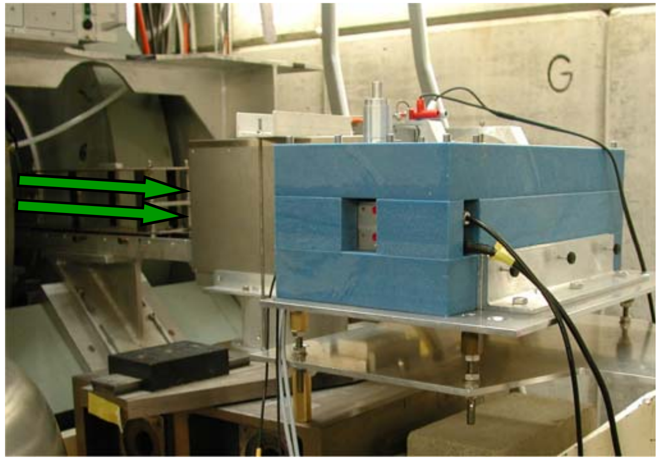
$\omega_{Larmor} = \omega_0 + \omega^*$

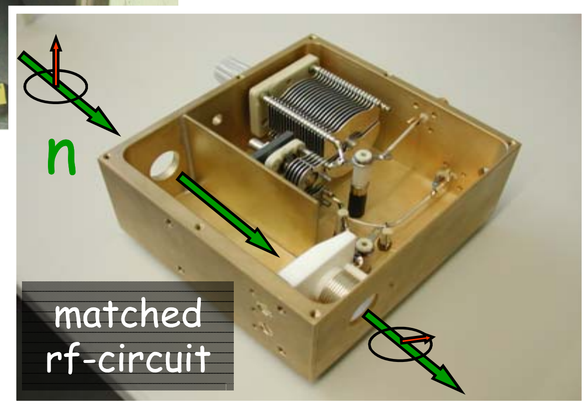
Spin-dependent scattering length

Pseudomagn. precession angle: $\varphi^* = \omega^* t \propto d\lambda \cdot b_i \cdot \frac{N}{V} P$

Ramsey-setup

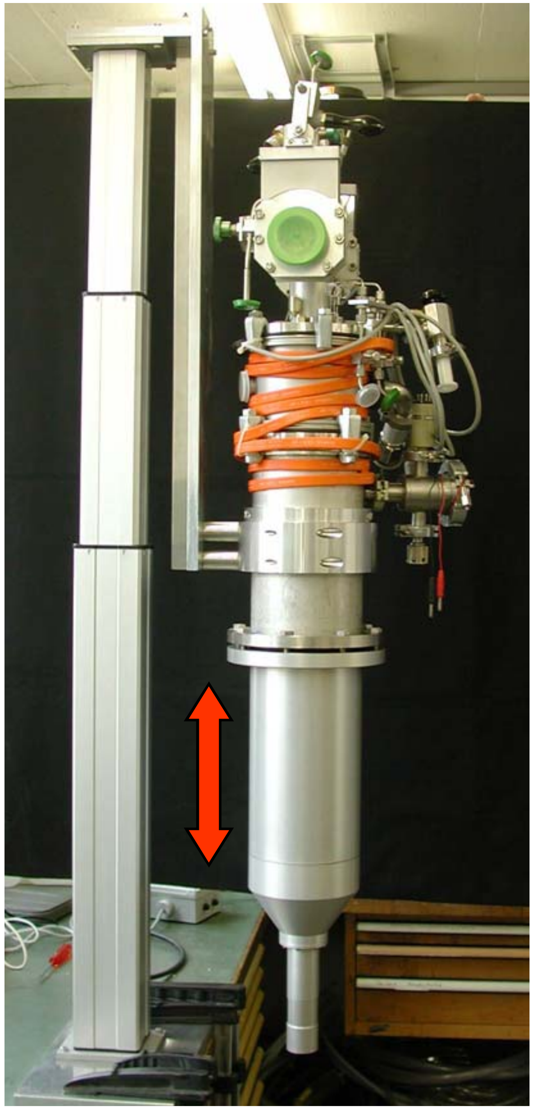






Neutron flight-path @ FUNSPIN-SINQ

cryostat / frozen spin target



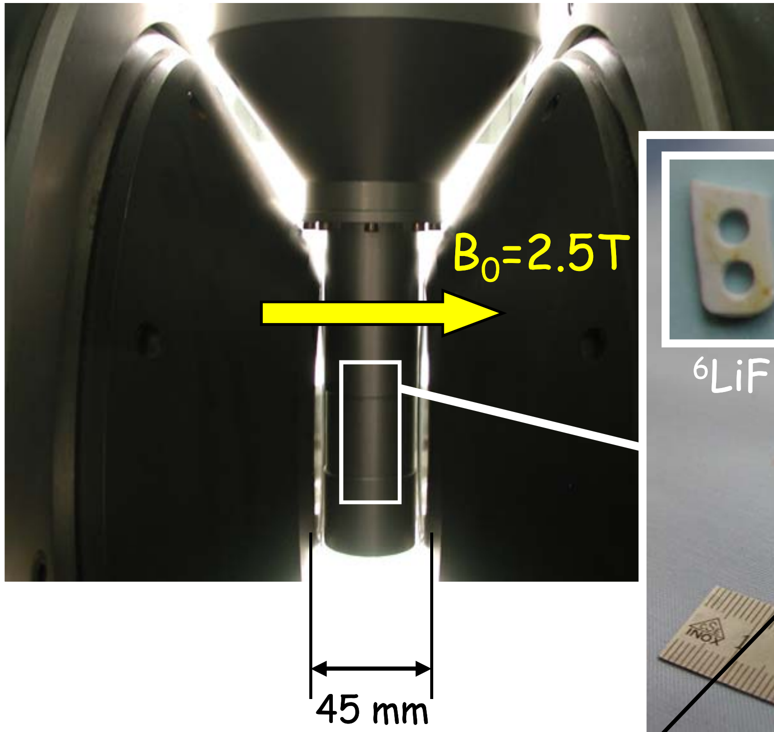
Requirements:

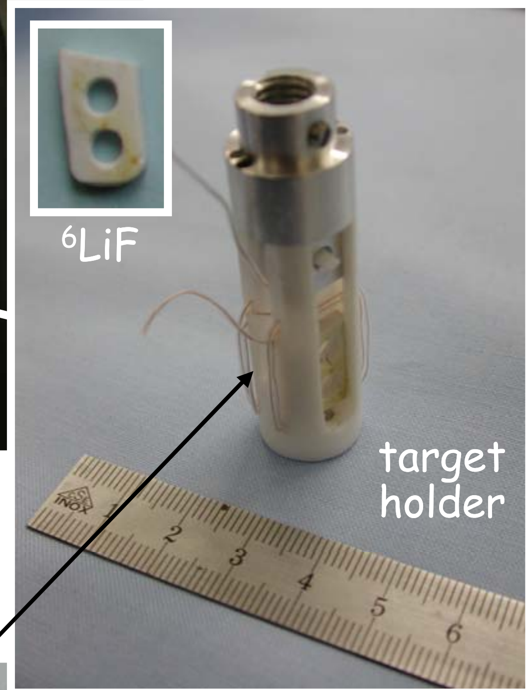
- Low temperatures to produce nuclear polarisation (DNP) and to avoid nuclear spin–relaxation and cross-relaxation
- No ^{3}He in the neutron beam path (absorption)
- Measure the nuclear polarisation (NMR)

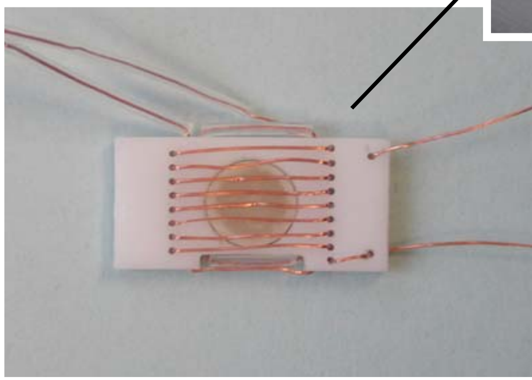
Solutions:

- Dilution refrigerator for frozen spin mode operation with large cooling power ($dQ_c/dt \sim 1$ mW at $T \sim 100$ mK)
- Target cell separated from mixing chamber and filled with L^{4}He and cooling via a silver sintered heat-exchanaer

cryostat & target



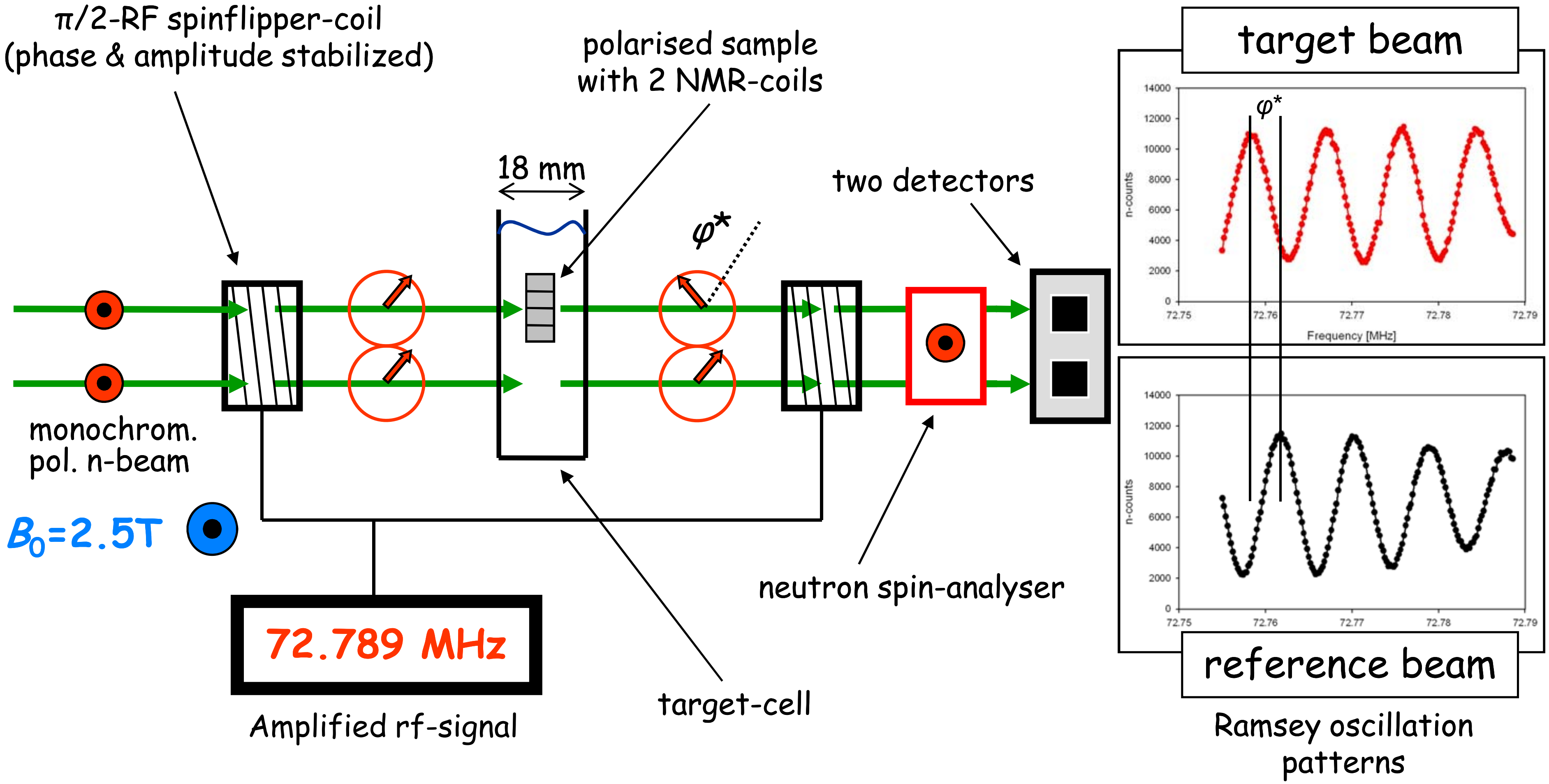




d-PS target
doped with d-TEMPO

Ø 5 mm x 1.2 mm

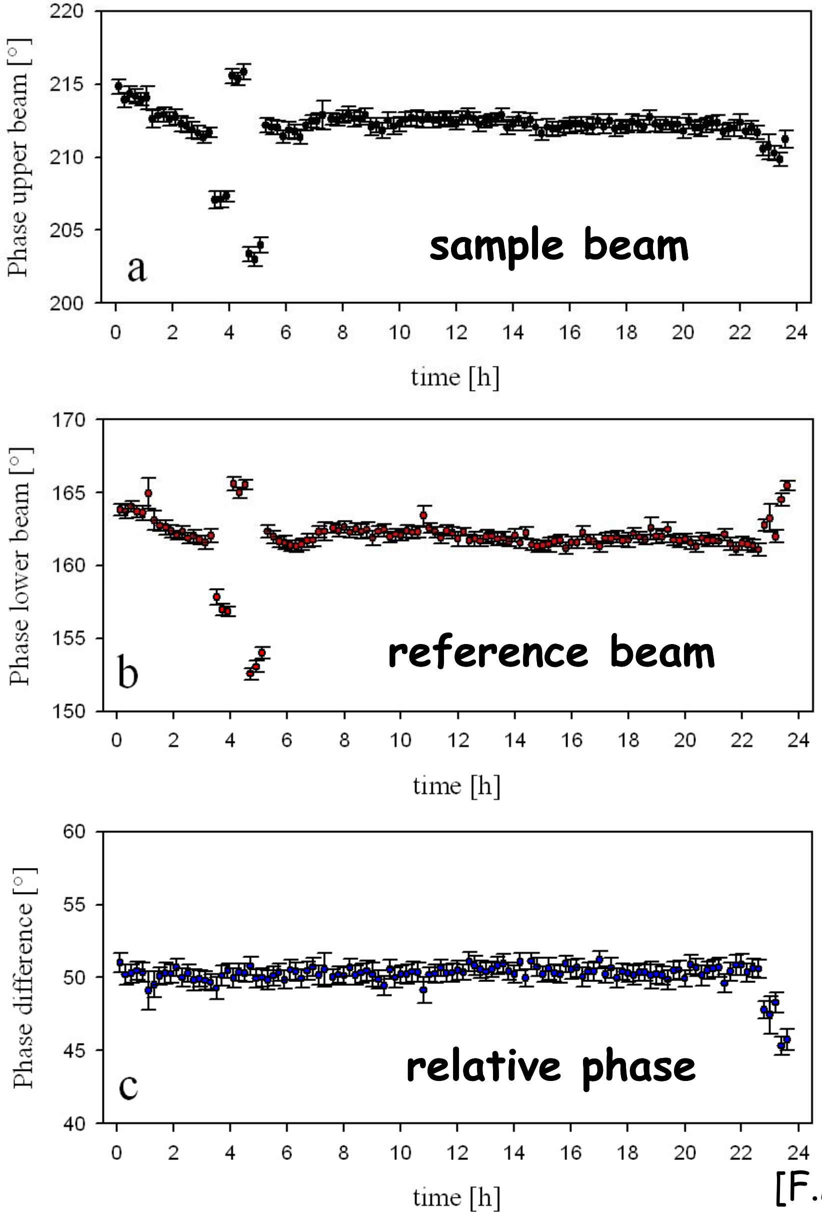
two-beam method / frequency scan



In 10 cm and at 2.5 T the neutron spin precesses 7500 times !!!

phase retrieval ~ ± 1° ⇨ typ. 1000° ⇨ 10^{-3}

test of the phase stability



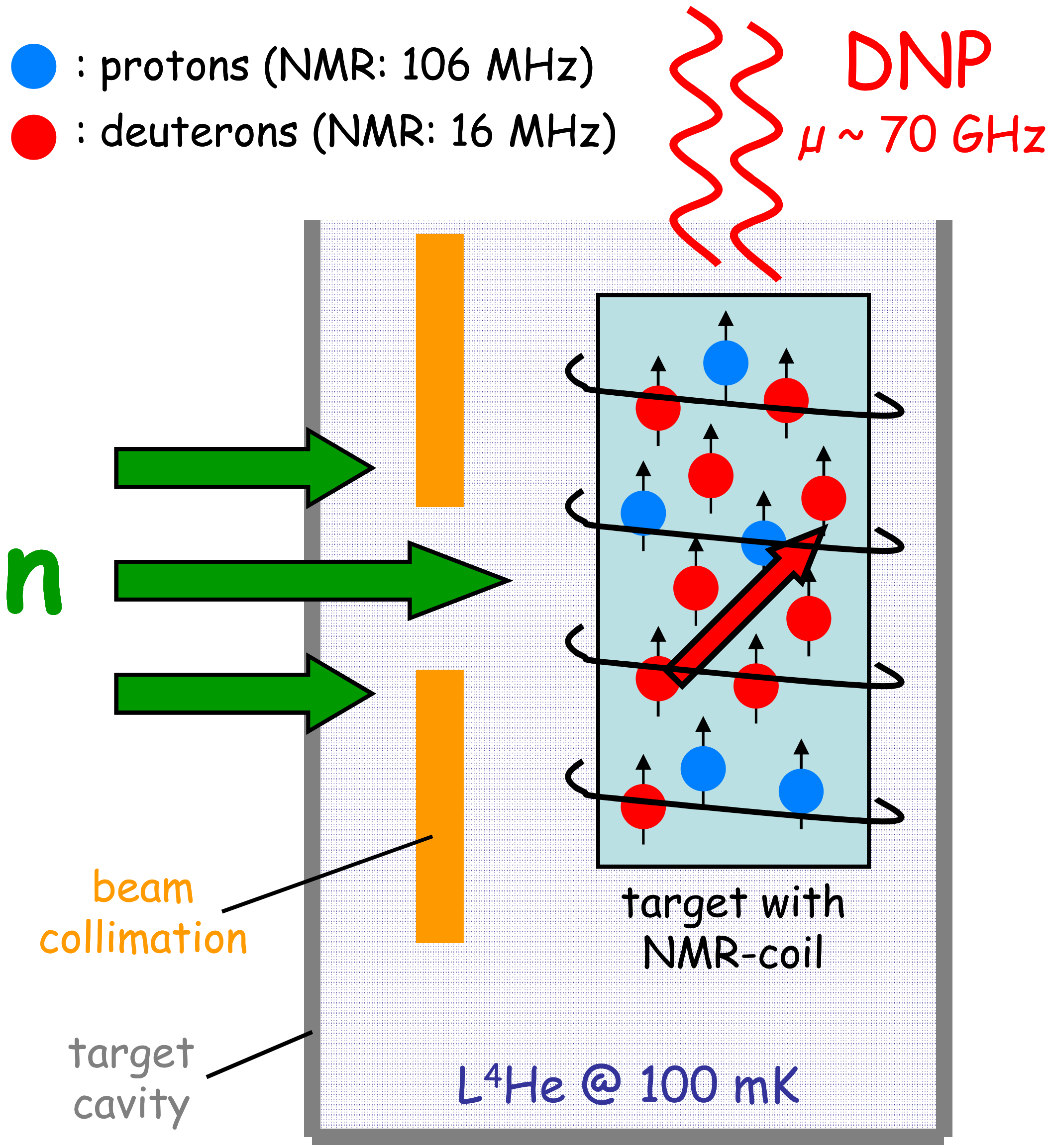
magnetic field: ± 0.3 ppm @ 2.5 T
spin flippers: ± 0.2° @ 73 MHz

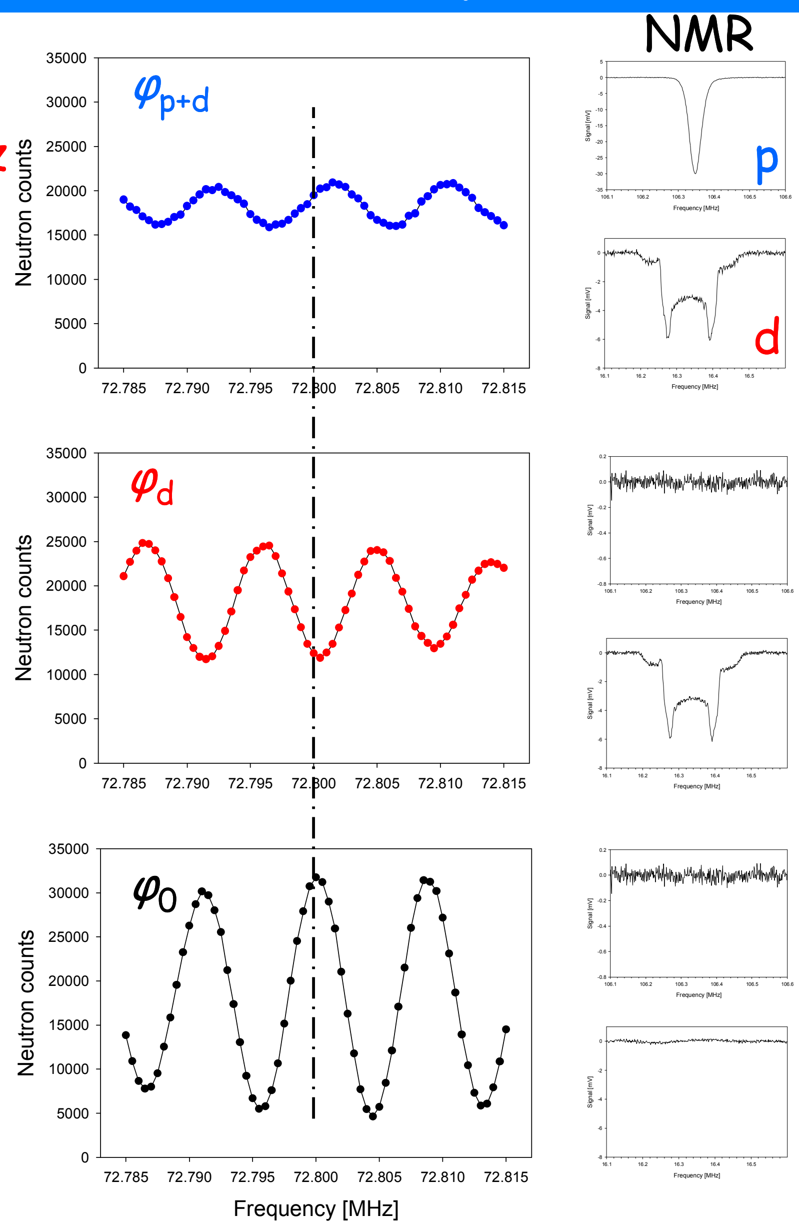
stabilised with feedback-loops

Relative phase stability better than ±0.4°

[F.M. Piegsa et al., *Nucl. Instr. Meth. A* **589** (2008) 318]

measuring procedure



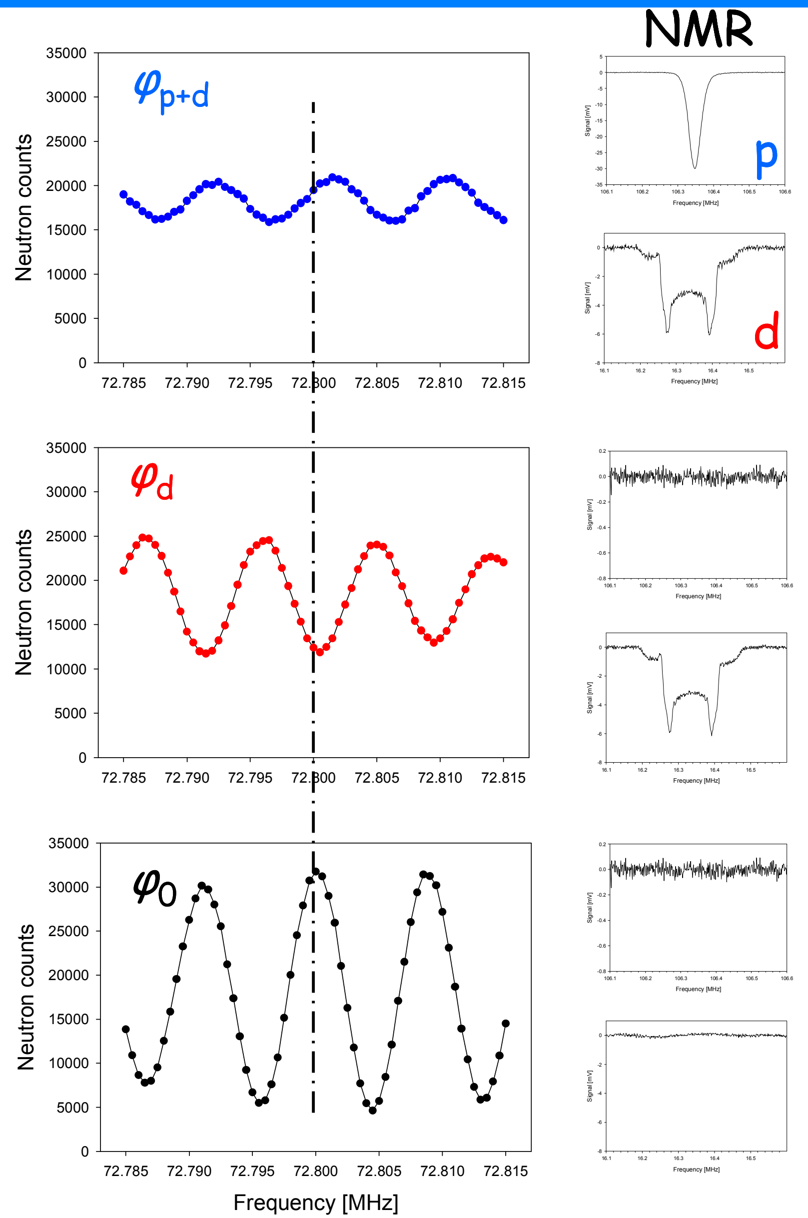


measuring procedure

$$b_{i,d} \propto b_{i,p} \cdot \frac{I_{NMR,p}}{I_{NMR,d}} \cdot \frac{\varphi_d - \varphi_0}{\varphi_{p+d} - \varphi_d}$$

Relative measurement !

No absolute values are needed !

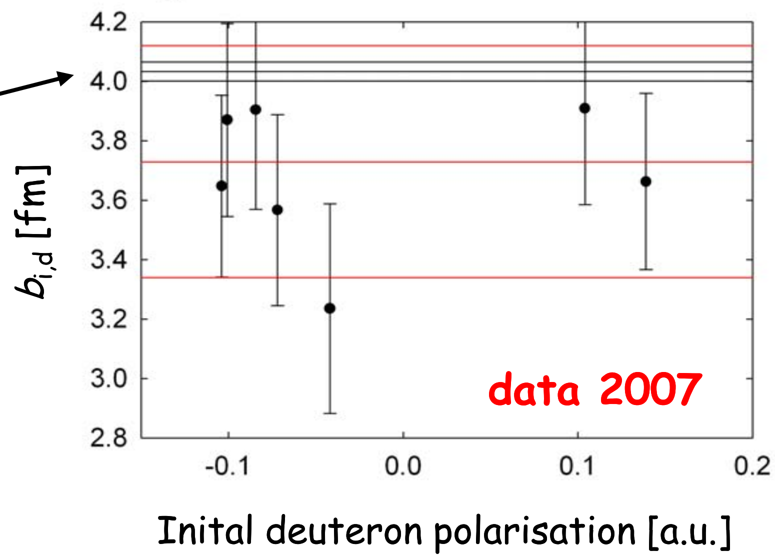


results & limitation in 2007

$b_{i,d}$ = (4.033 ± 0.032) fm

$$\sigma_{tot} = 4\pi(b_c^2 + b_i^2)$$

[Dilg et al., PLB **36** (1971) 208]

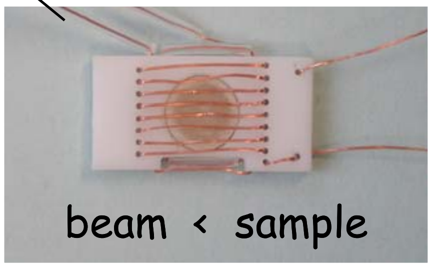


$b_{i,d}$ = (3.73 ± 0.05 ± 0.06 ± 0.28 ± ?) fm

stat. uncertainty of **NMR** and the **pseudomagn. phase shift** measurement

stat. and syst. uncertainty of the **NMR cross-calibration**

(ramping of magn. field from 2.5 T to 0.4 T)



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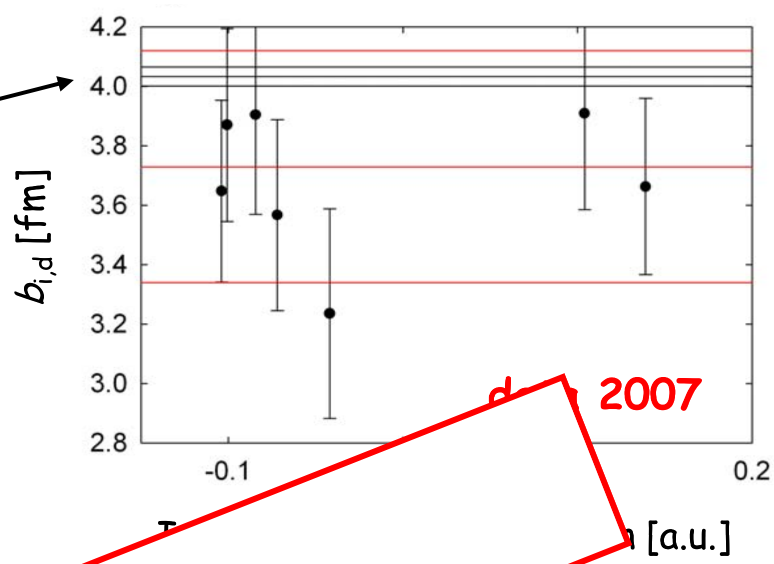
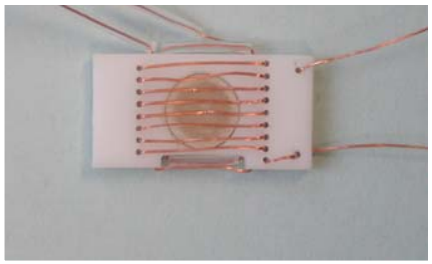
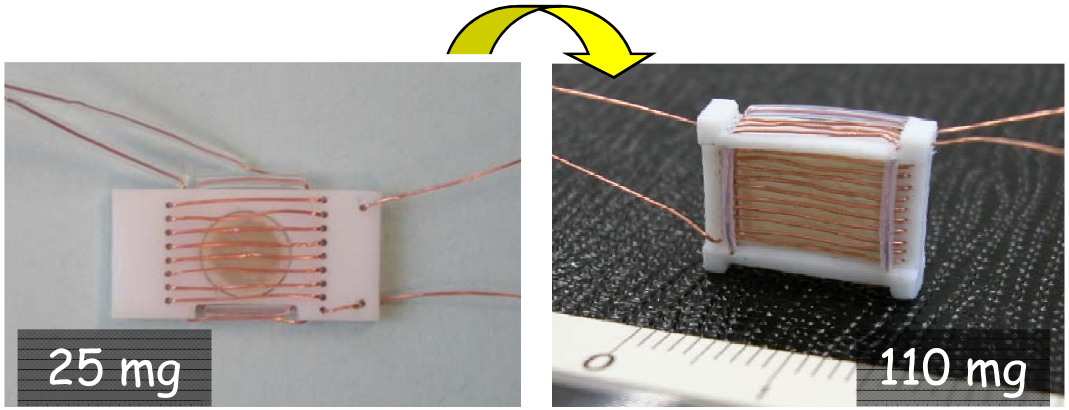


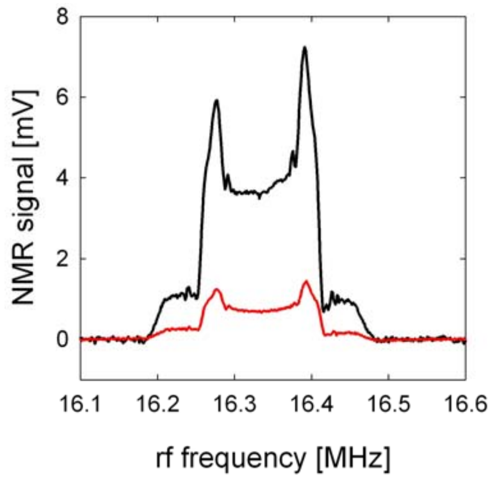
$b_{i,d}$ = (3.73 … .28 ± ?) fm

Limited by NMR & Spin Relaxation



the obvious solution: use a larger sample

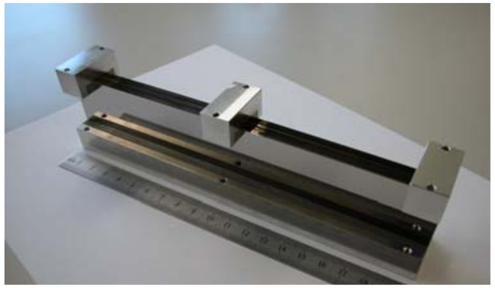




Larger NMR signals by factor 4, but …

… but new/old problems

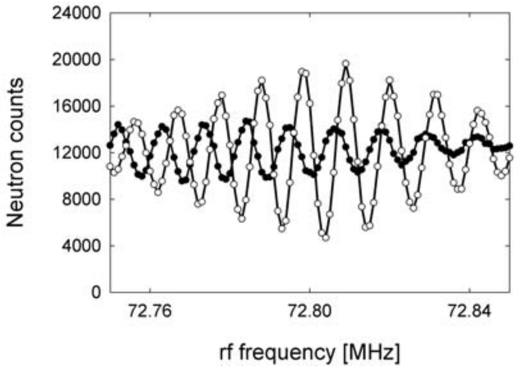
1. Beam collimation:



F.M. Piegsa,
NIM A **603** (2009) 401

FWHM ~ 0.1°
Transmission ~ 80% @ 5 Å

2. Inhomogeneity of the magnetic field and sample:



3. What remains: Spin Relaxation at low fields – Cross-calibration.

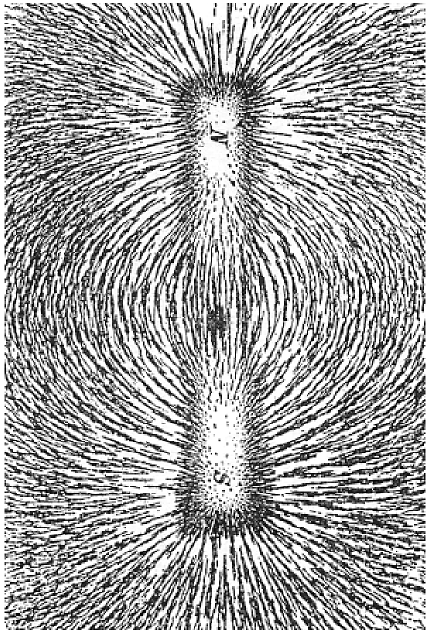
Ramsey-method is an elegant way to measure $b_{i,d}$,
but at the moment it is systematically limited.

Neutron Spin Phase Imaging

a „spin-off“ project

F.M. Piegsa, B. van den Brandt, P. Hautle & J.A. Konter

Imaging of magnetic fields …

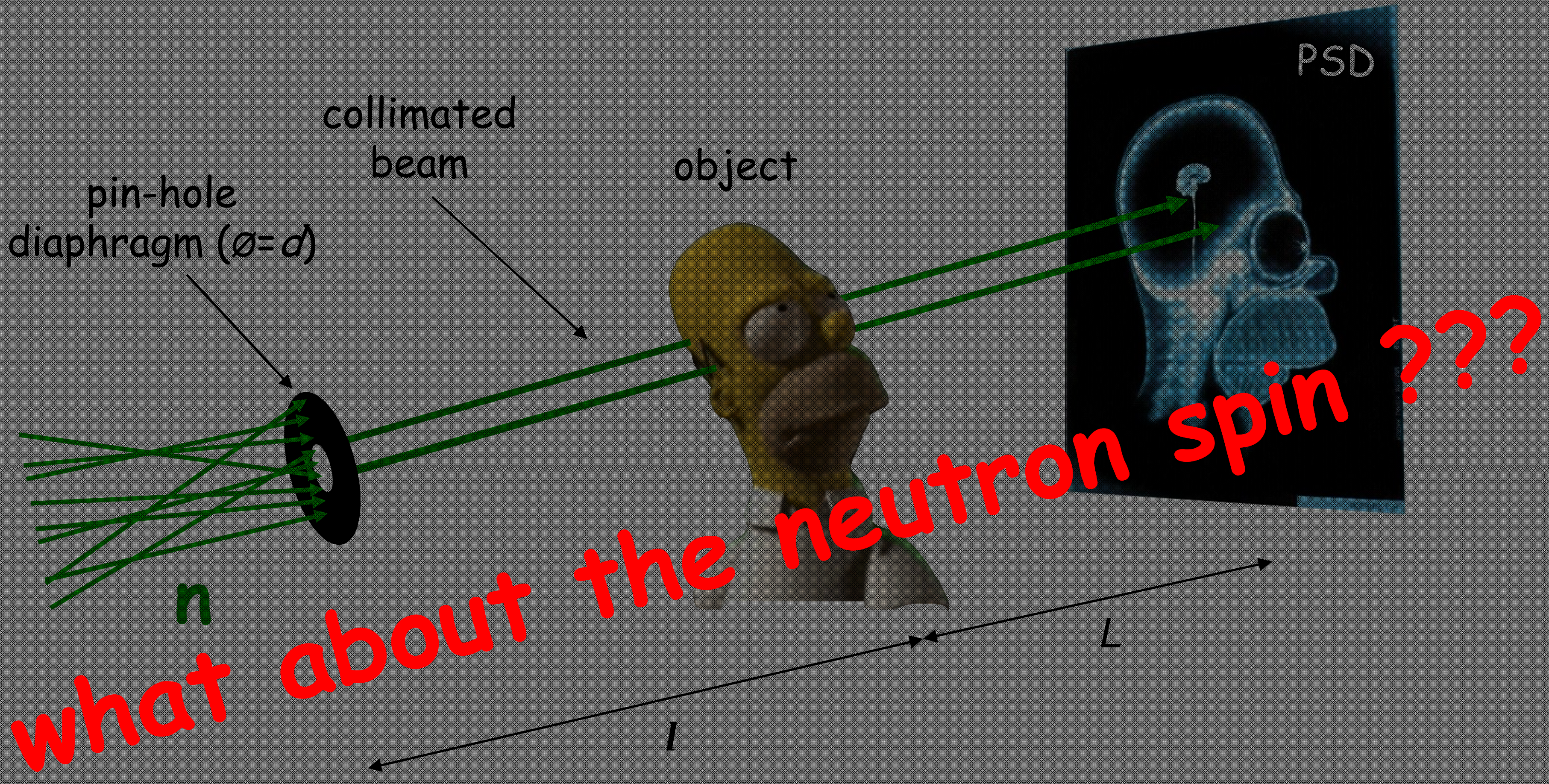


… with iron powder and …

… with neutrons.

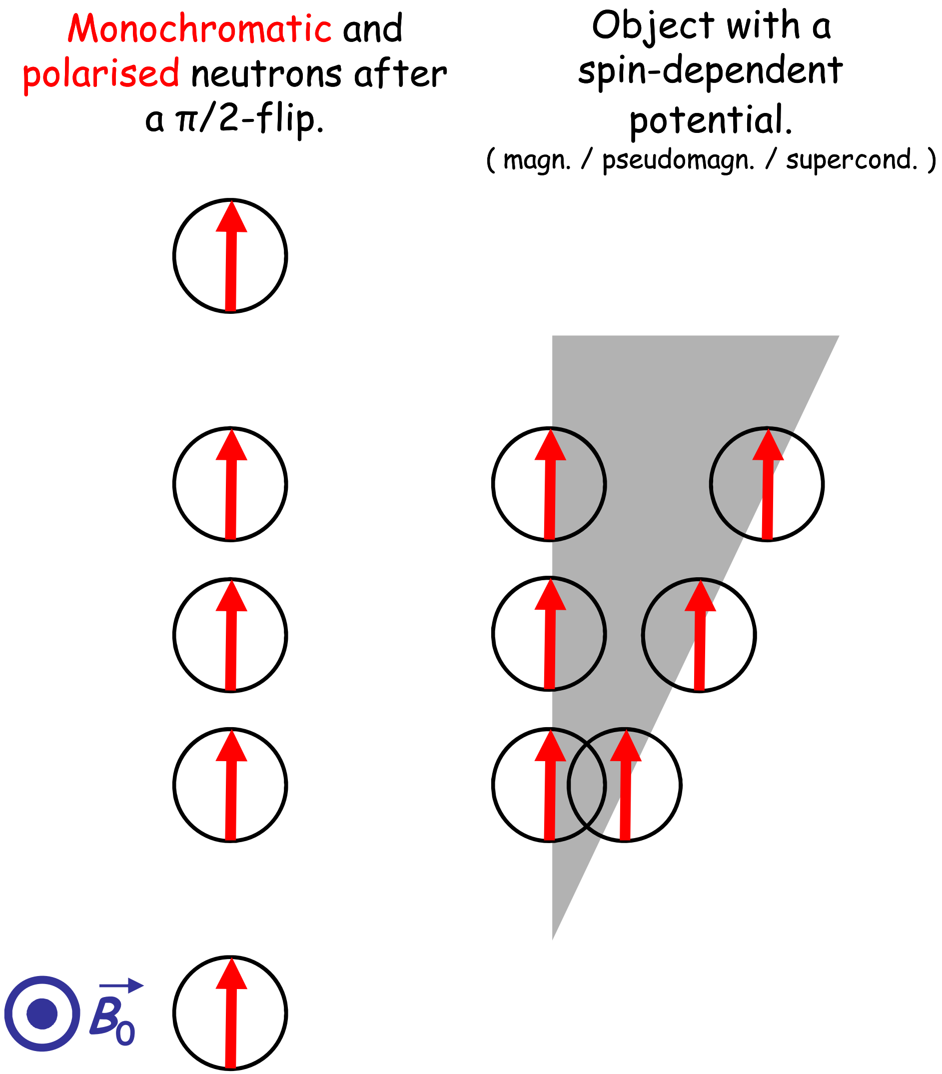
[F.M. Piegsa et al., *Phys. Rev. Lett.* **102** (2009) 145501]

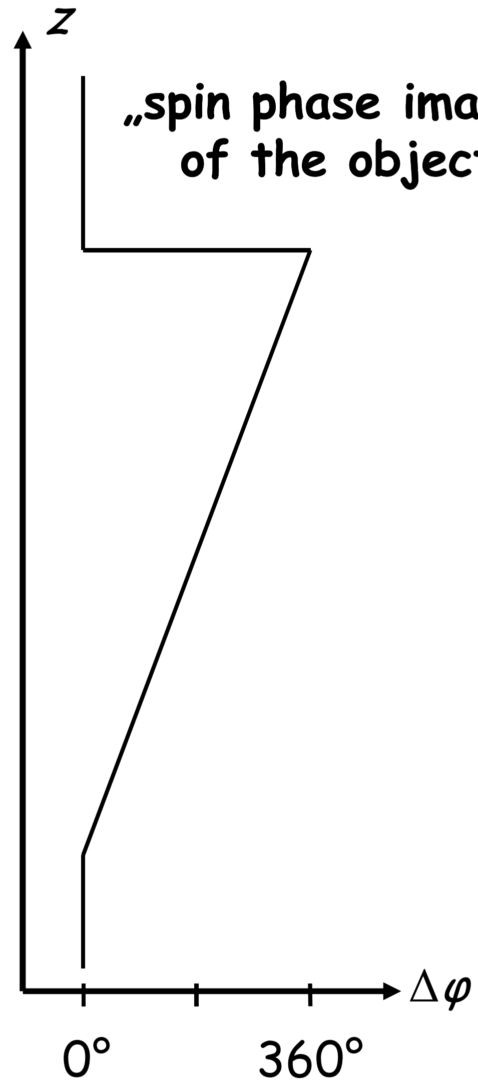
standard neutron imaging principle



- imaging principle relies on **beam attenuation** ($\sigma_{tot} = \sigma_{scatt} + \sigma_{abs}$)
- **unpolarised**, **white** thermal / cold neutrons

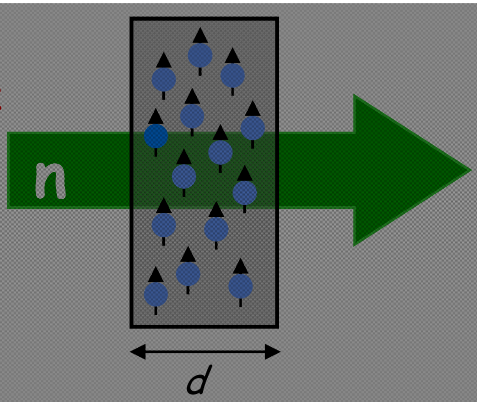
imaging principle by neutron spin precession





how large are the phase shifts ?

Pseudo-magnetic



- **thermal equilibrium polarisation:**

$$P \propto \gamma_{\text{nucl}} \cdot \frac{B}{T}$$

e.g. @ 1T & 1K:
protons: **0.1%**

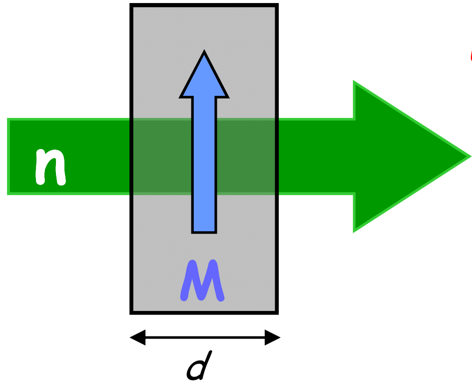
- **pseudomagn. precession angle:**

$$\varphi^* = 2\lambda d \sqrt{\frac{I}{I+1}}\, N\, P\, b_i$$

λ = 5 Å, N_{proton} = 80 mol/l, P = 0.1%

⇨ $\varphi^* \approx$ **40°/ mm**

Magnetic



- **„objects" to image:**
  - ferromagnetic materials
  - superconductors ($T < T_c$)
  - magnetic fields

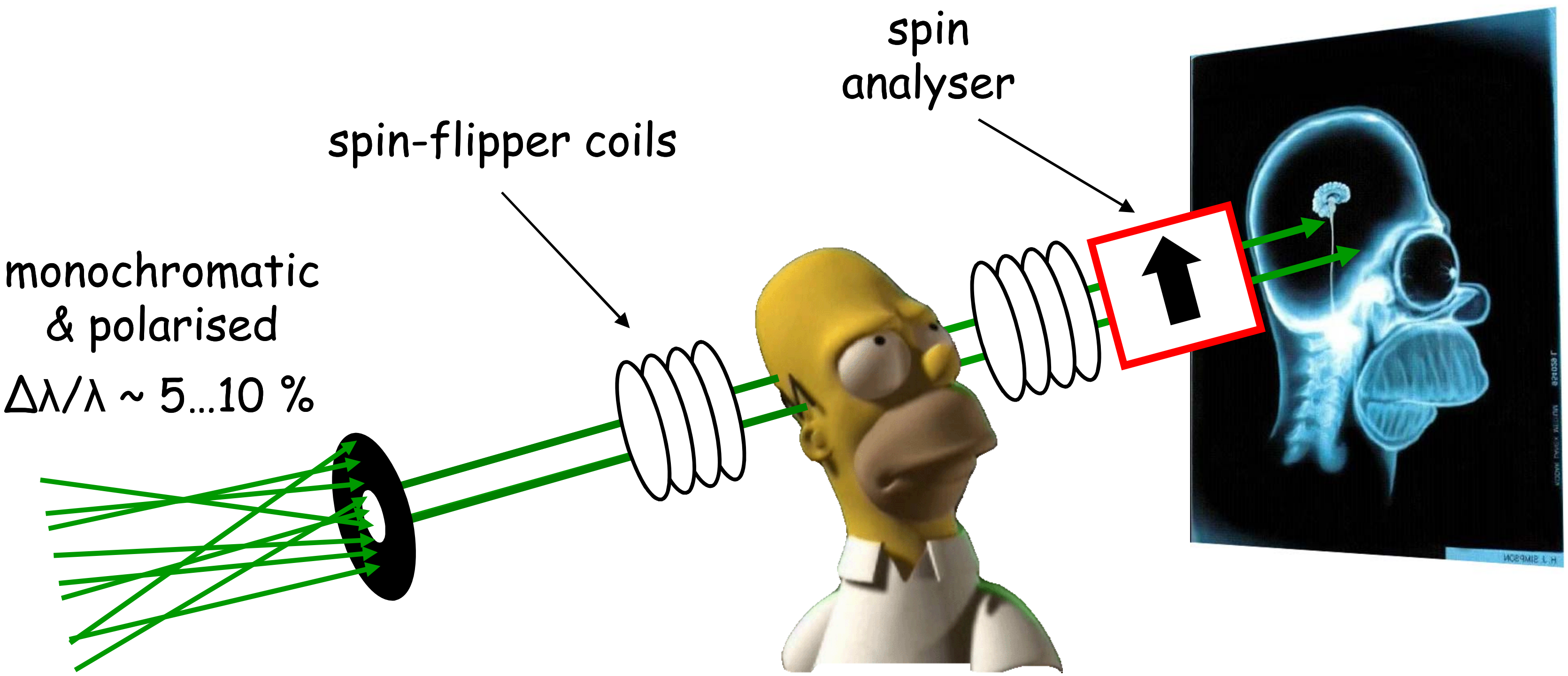
- **magnetic precession angle:**

$$\varphi_m = \lambda d m_n \cdot \frac{\gamma_n B}{h}$$

For λ = 5 Å:

⇨ $\varphi_m \approx$ **13°/ µm T**

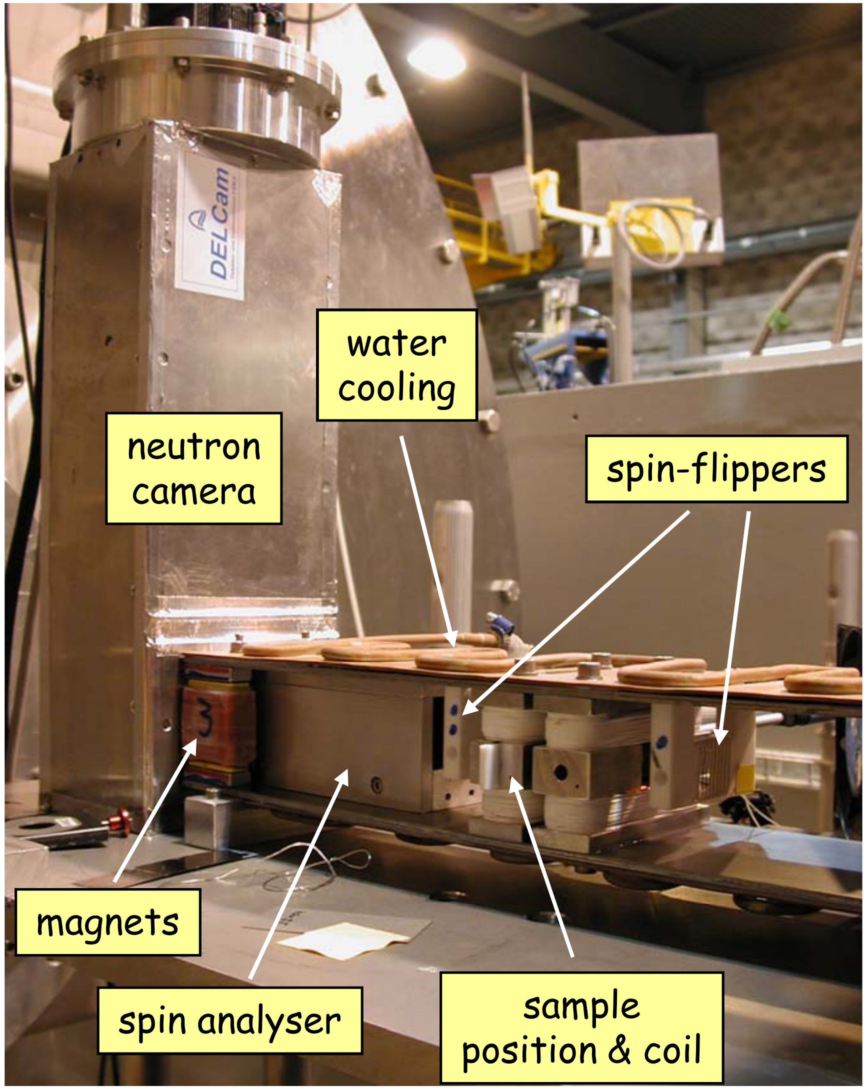
Combination of Ramsey's method with neutron imaging:



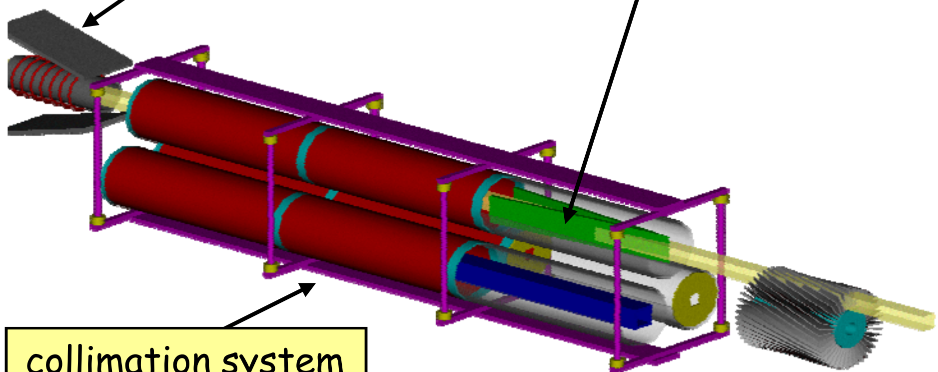
⇨ **Simultaneous „Attenuation" & „Spin phase" imaging !**

[N. Ramsey, *Phys. Rev.* **78** (1950) 695]
[F.M. Piegsa et al., *Nucl. Instrum. Meth.* A **586** (2008) 15]

NSPI at SANS-I (PSI)

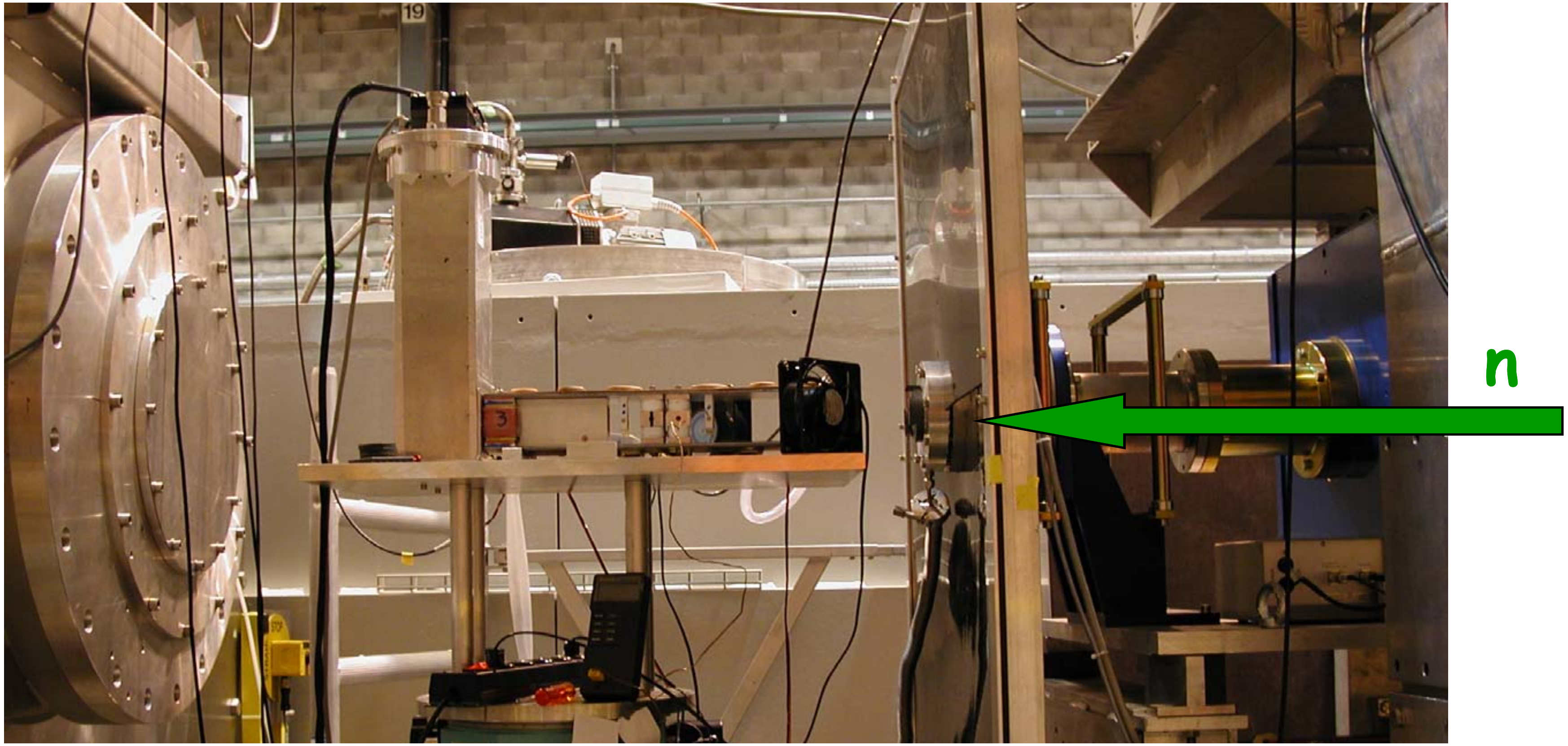


SANS-I:	l/d ≈ 280 Δλ/λ ≈ 10 %
Resolution:	0.8 mm (FWHM-PSF)
Sensitivity:	±7.5 x 10^{-8} Tm
Expos. time:	1 min/image x 11
Sample field:	5 … 30 mT (!!)



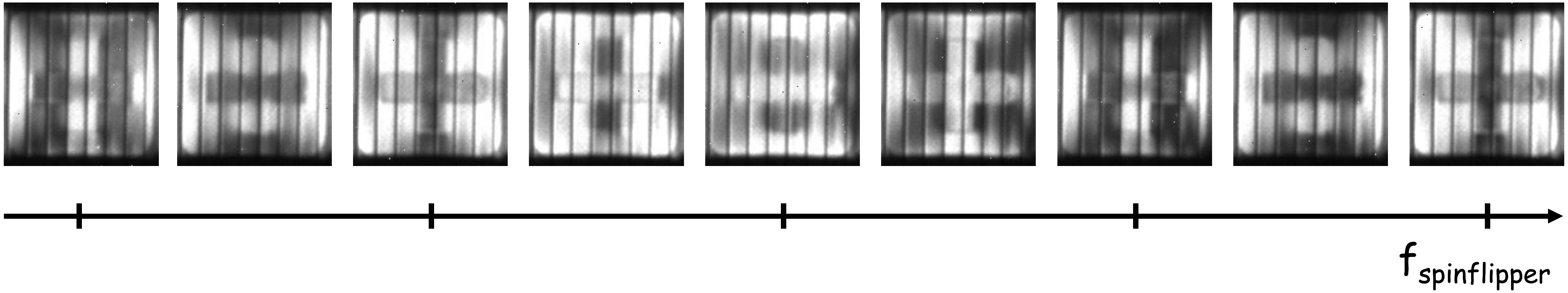
[Pol. SANS: V.K. Aswal et al., *Nucl. Instr. Meth. A* **586** (2008) 86]
[Setup: F.M. Piegsa et al., *Nucl. Instr. Meth. A* (2009) accepted]
[Analyser: F.M. Piegsa & M. Schneider, *Nucl. Instr. Meth. A* **594** (2008) 74]

NSPI at SANS-I (PSI)

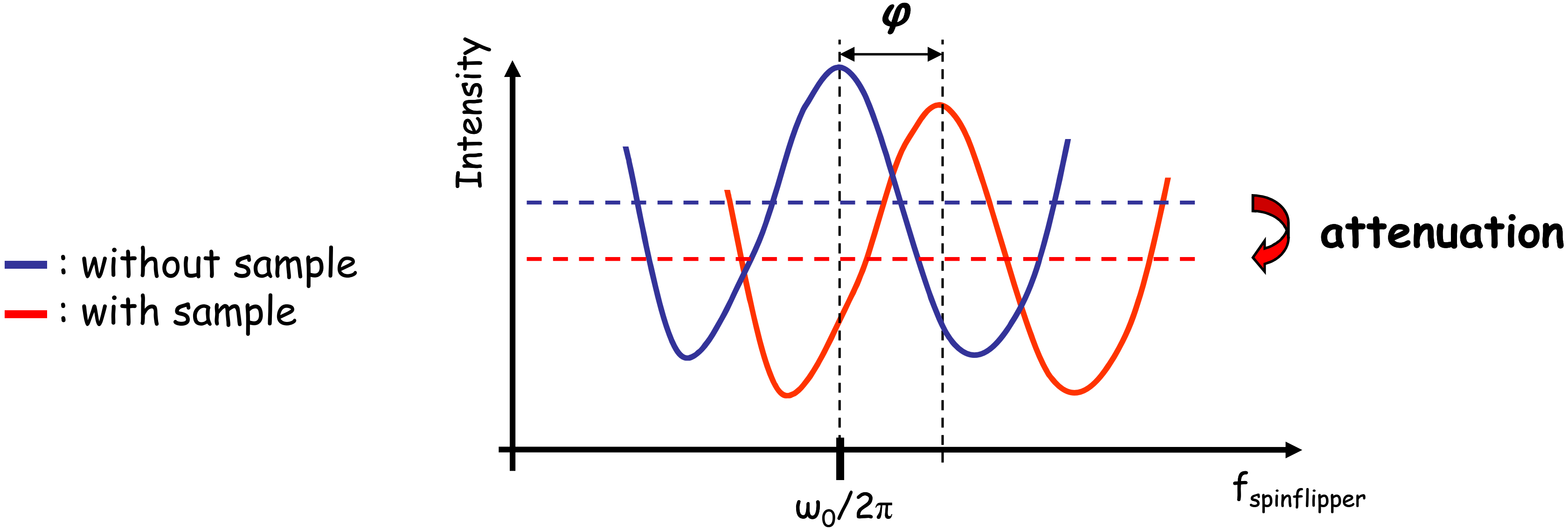


~ 700 mm

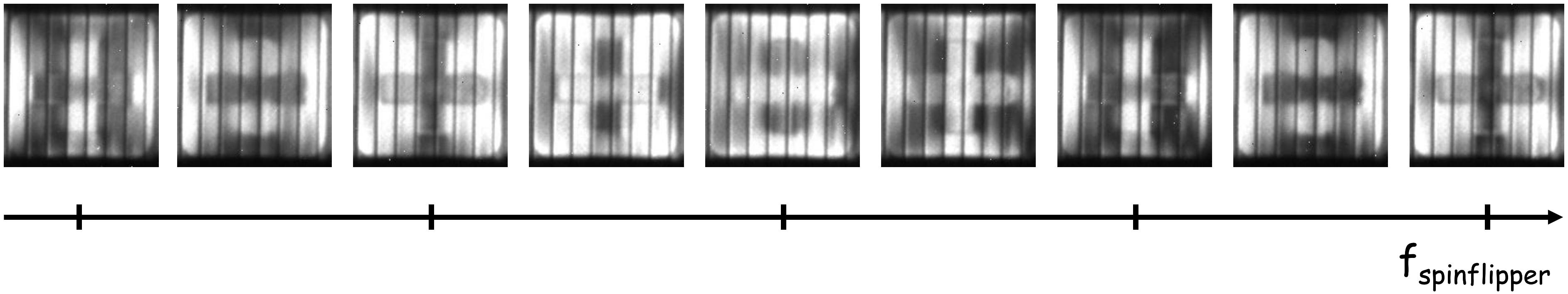
examples of the imaging method

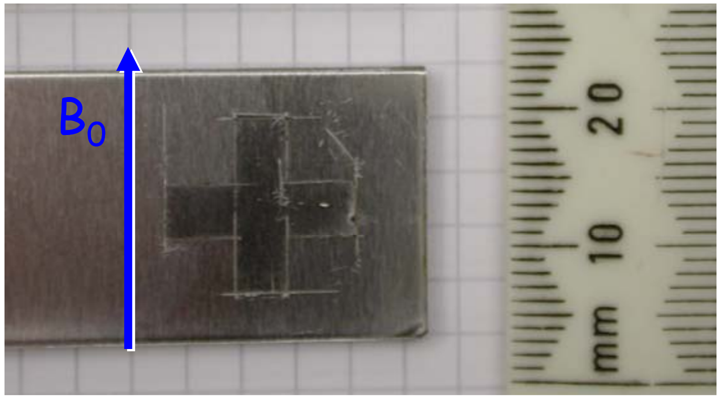


Analysis for each pixel:

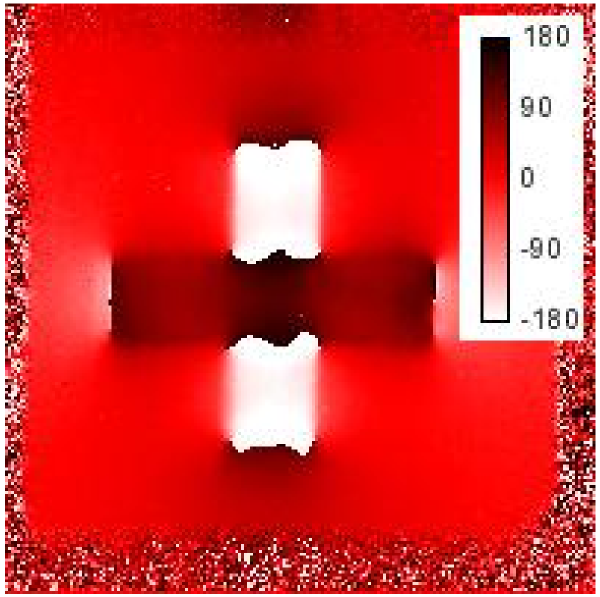


example I: the swiss cross





7.7 μm Fe sputtered on Aluminium



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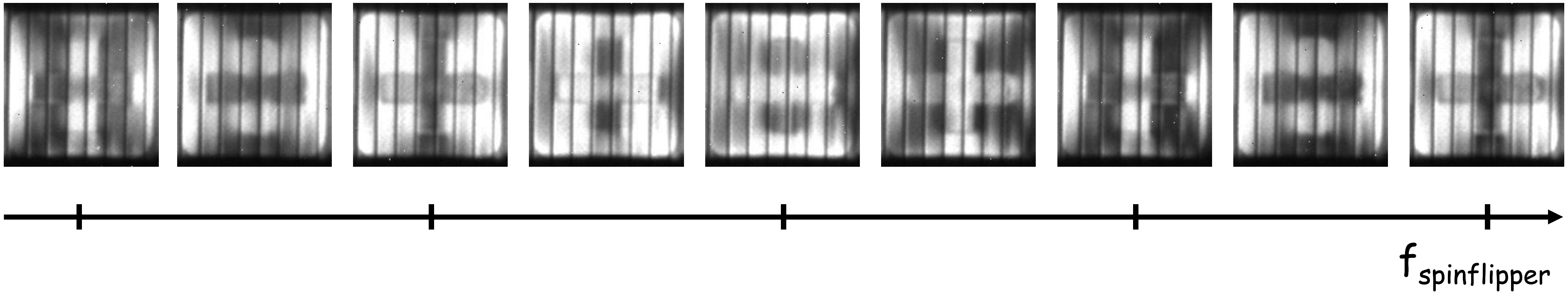
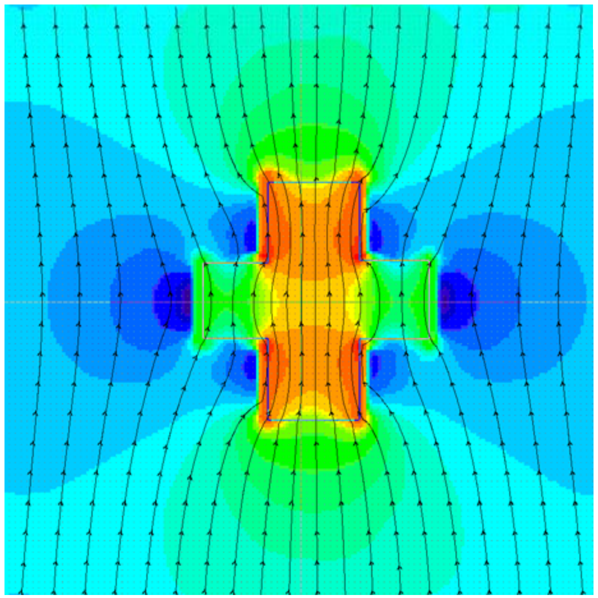
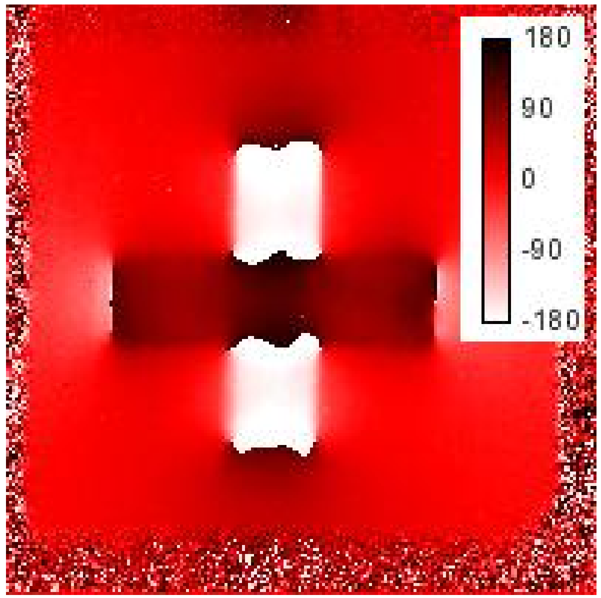
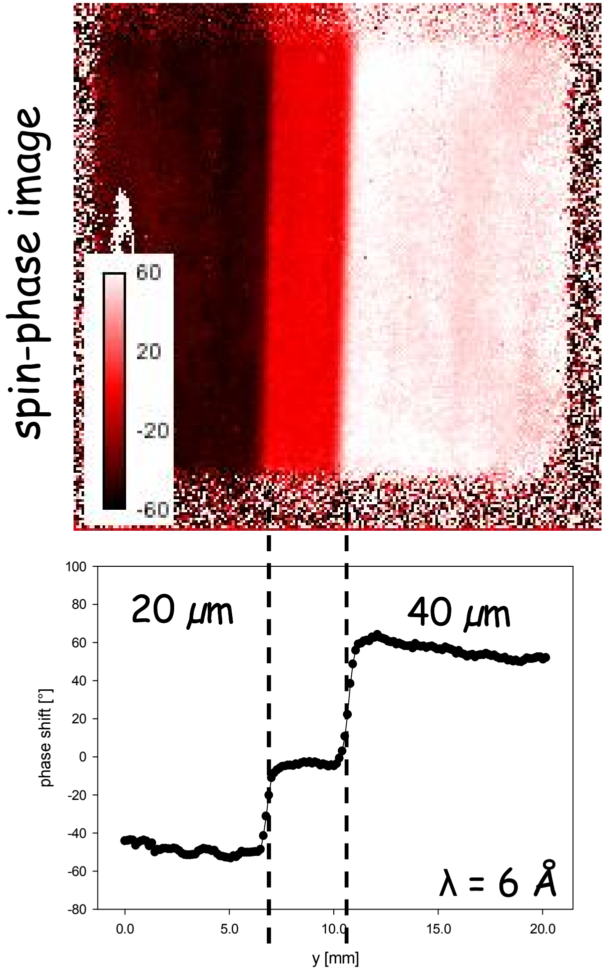


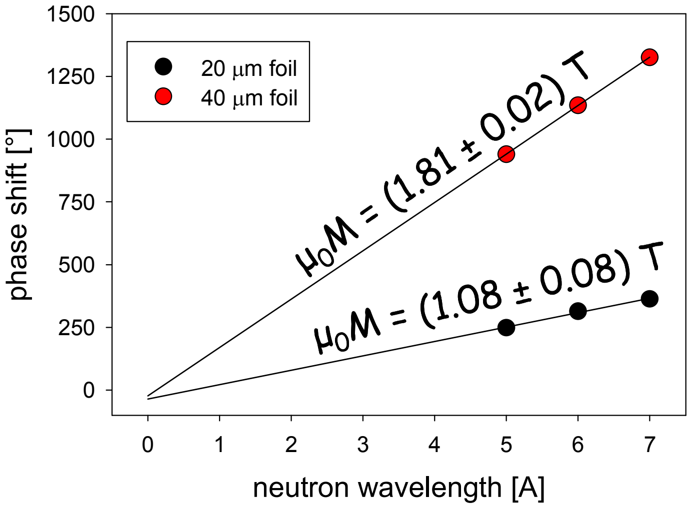


Qualitative simulation with 'Vizimag 3.15'



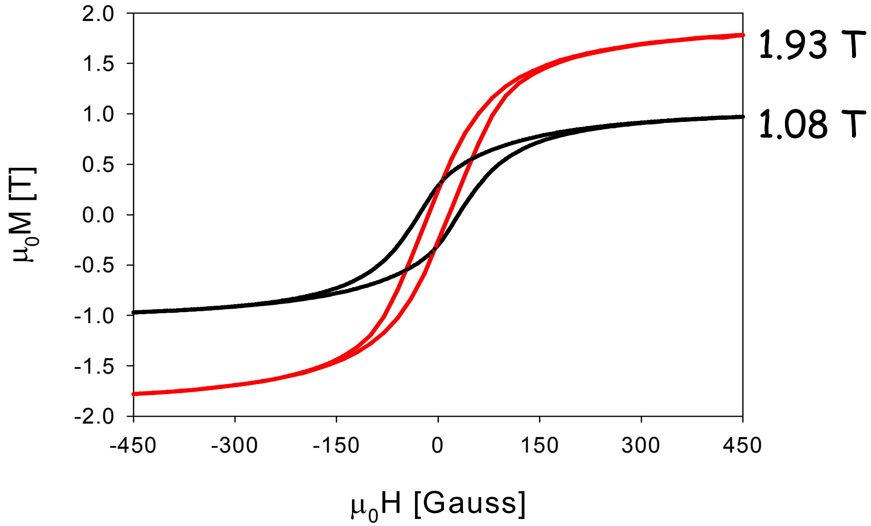
example II: precision shim steel foils

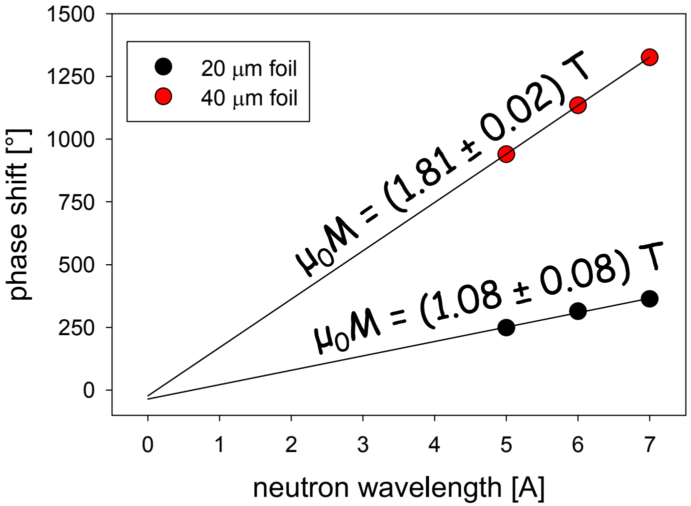




example II: precision shim steel foils

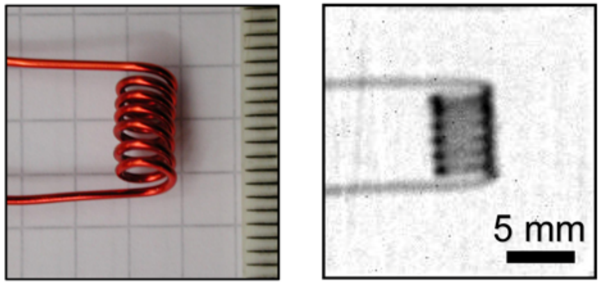
PPMS magnetisation measurement

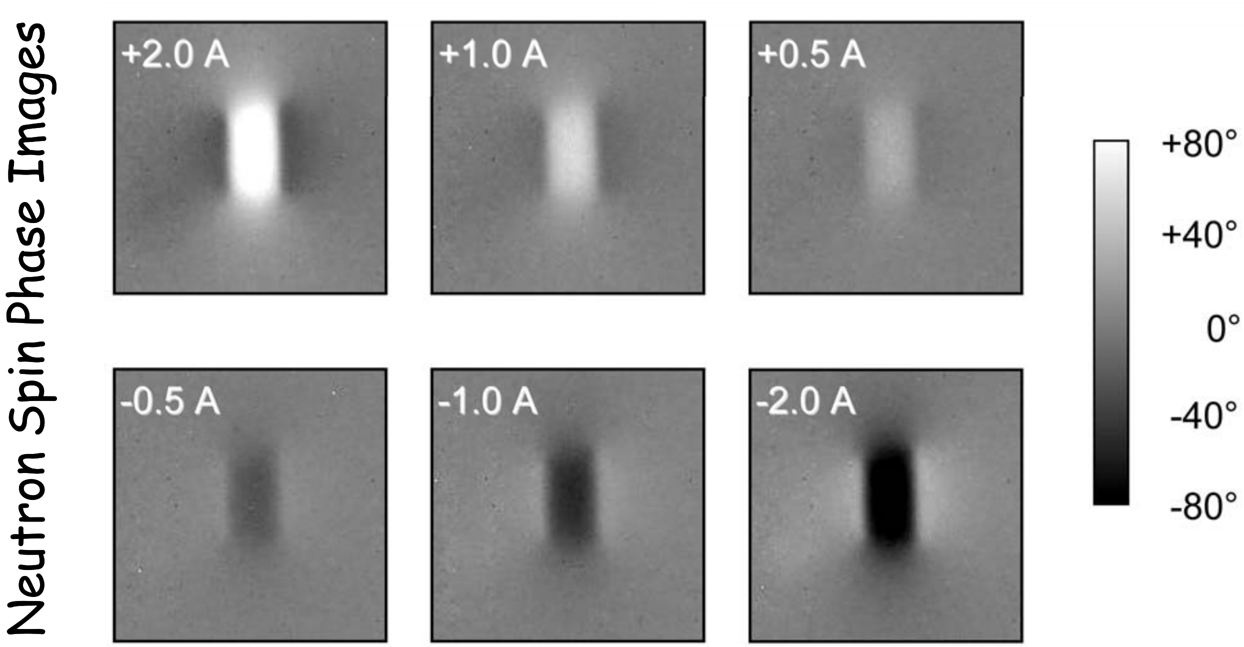




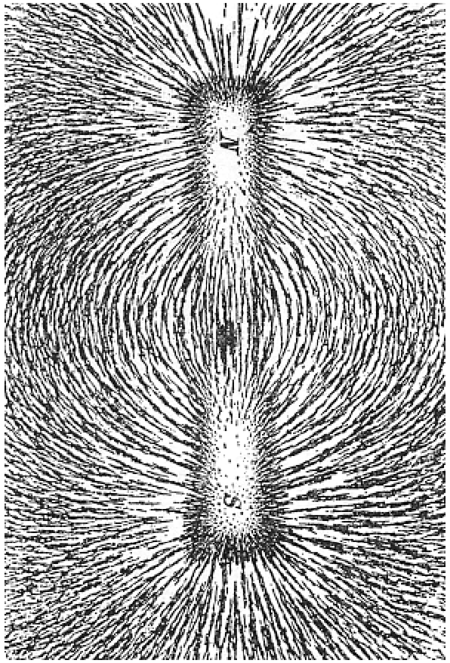
NSPI measurements are in good agreement with PPMS characterisations !

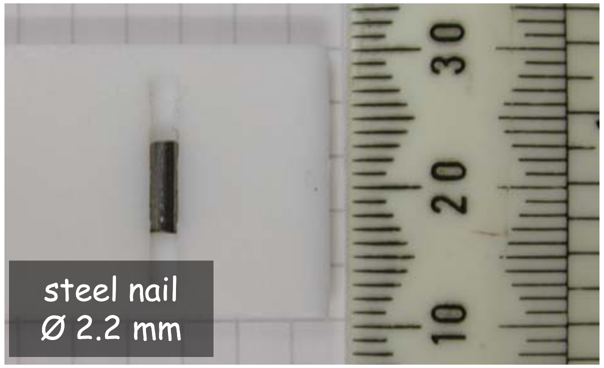
example III: magnetic field of a coil

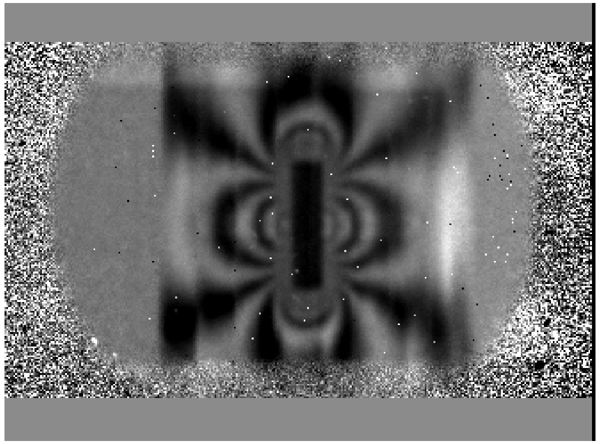




example IV: imaging of a dipolar field







… with iron powder and …

… with neutrons.

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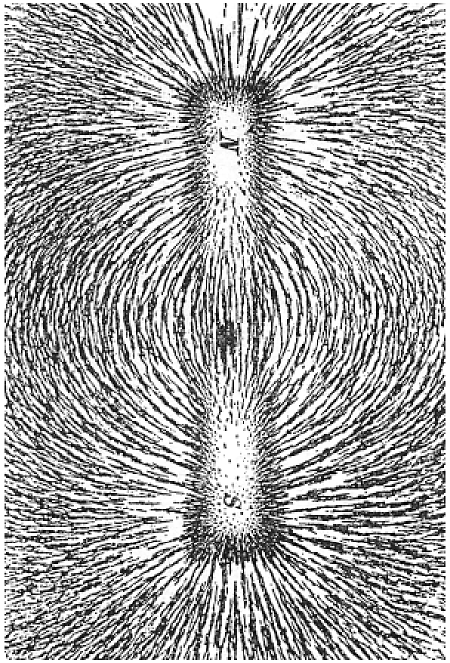
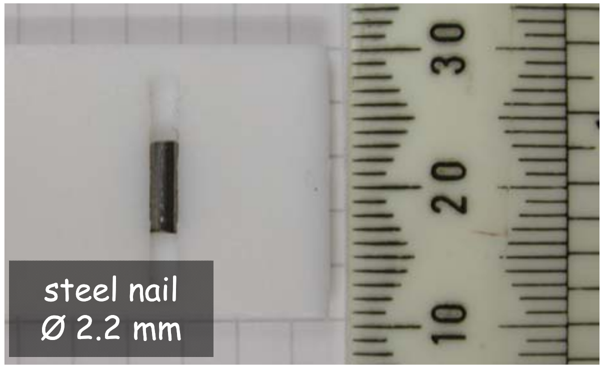
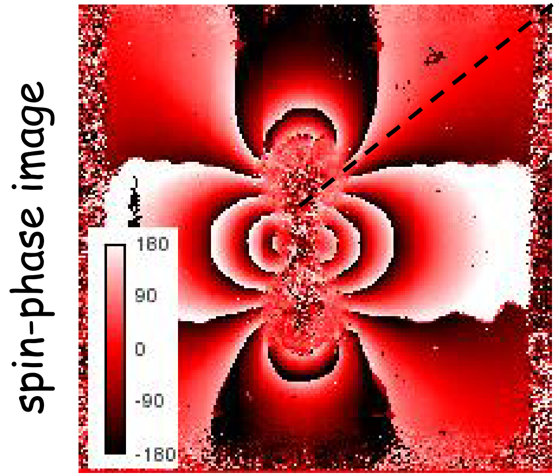


… with iron powder and …



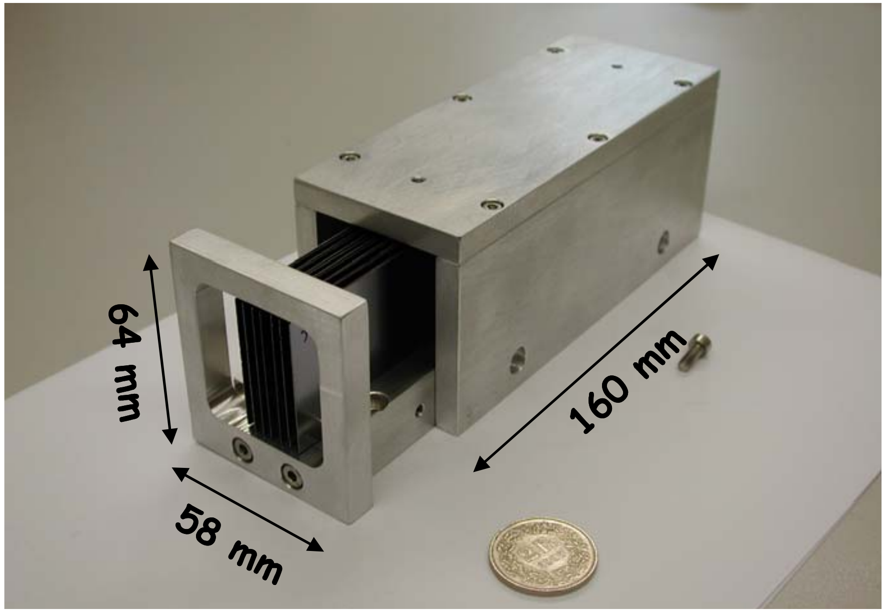


… with neutrons.

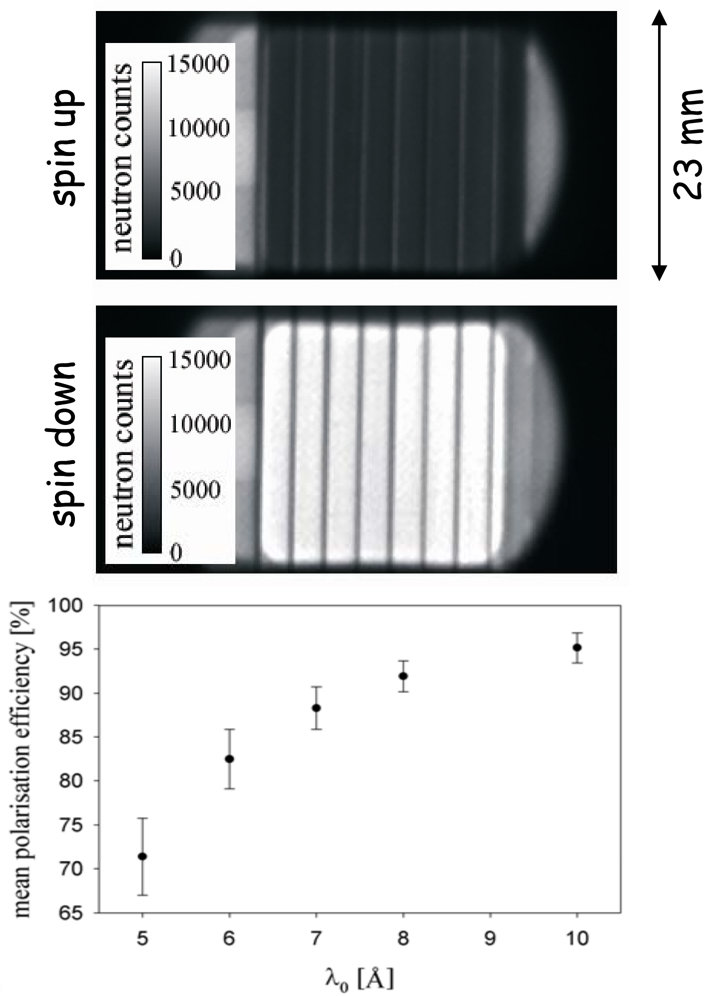
- An experiment with the goal to determine the spin-dependent neutron-deuteron scattering length using a target containing polarised nuclei.
- A novel quantitative neutron radiography technique sensitive to neutron spin interaction.
- In both cases the neutron spin precession is measured using the Ramsey technique.

Thank you for your attention.

a compact transmission polariser



2D images at λ=5Å at SANS-I

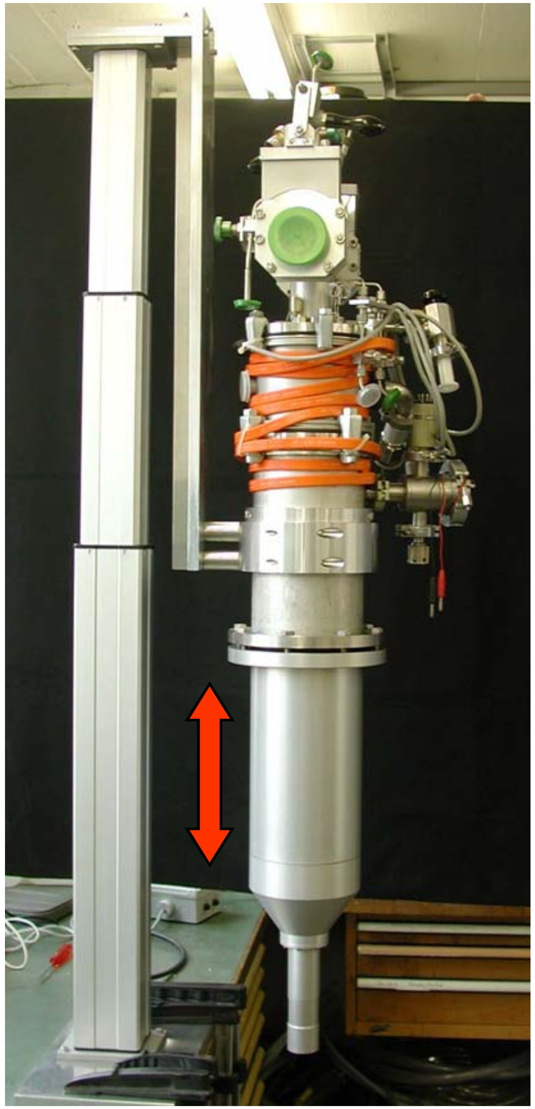


- **Analysing cross-section: 22 x 34 mm^2**
- **Transmission Polariser (*T*=44% @ 5 Å)**
- **No Reflected beam**
- **$Q = T P^2 = 0.27 \pm 0.03$**

  (equivalent to polarised ^{3}He gas *P*~75%)

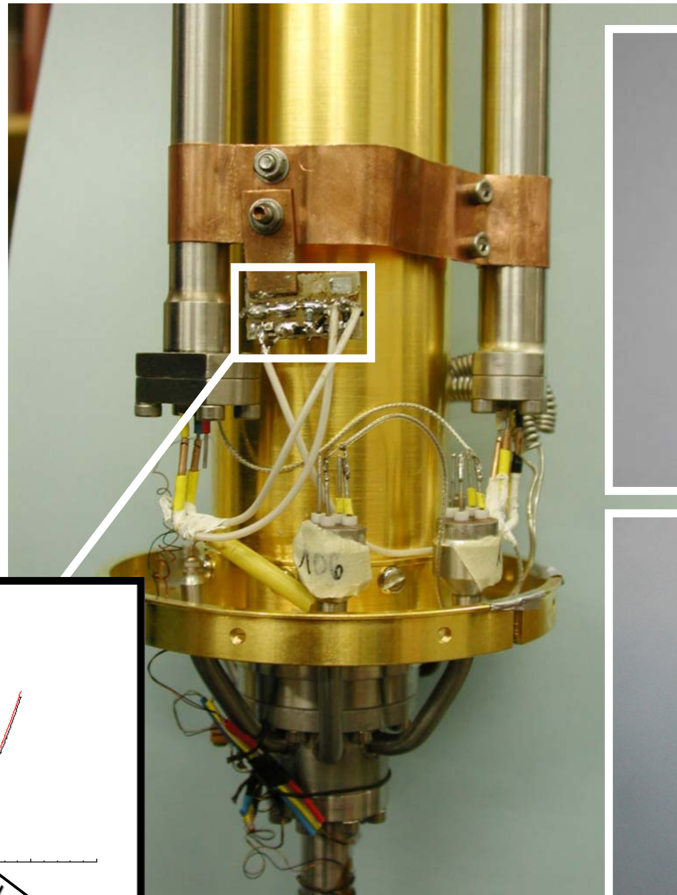
[F.M. Piegsa & M. Schneider, *Nucl. Instr. Meth.* A **594** (2008) 74]

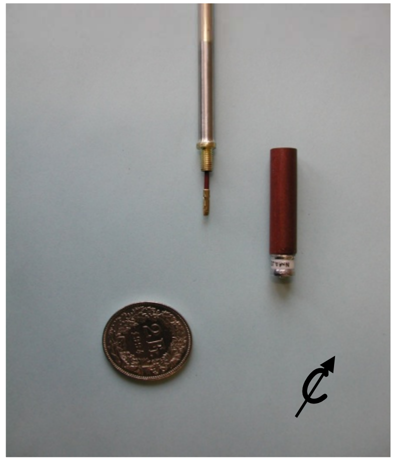
cryostat / frozen spin target



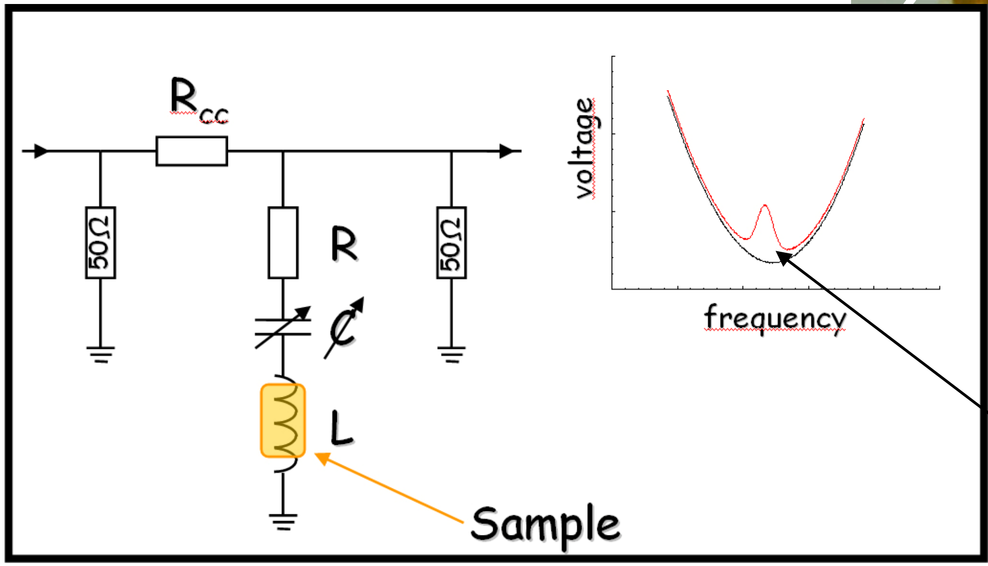
[P. Roubeau, *J. Phys.* **39** (1978) C6-1146]
[B. van den Brandt et al., *Nucl. Instr. Meth.* A **289** (1990) 526]

low temperature cw-NMR



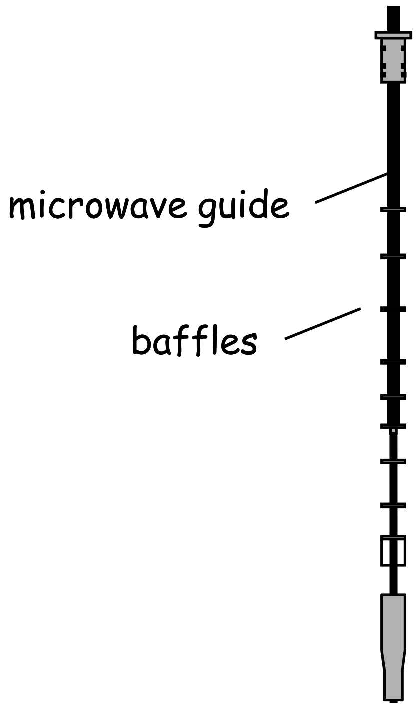


serial LCR-ciruit @ 1K

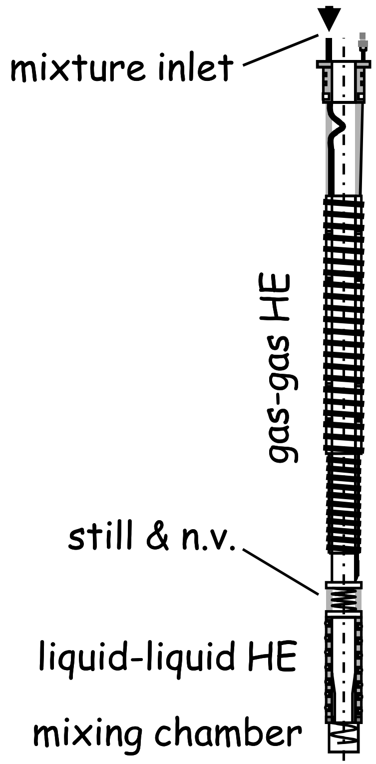


Nuclear Polarisation ∝ Area

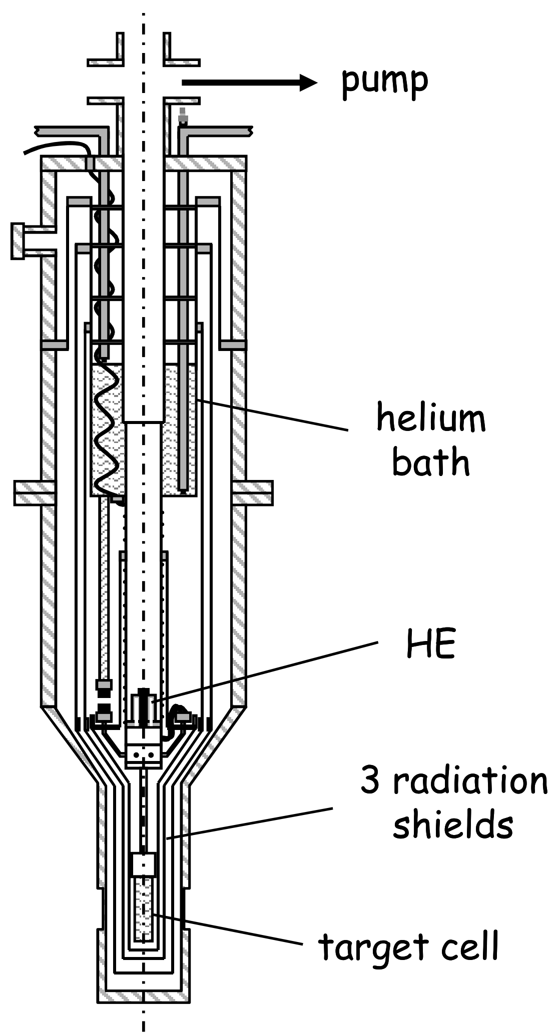
Roubeau type cryostat



sample/microwave insert



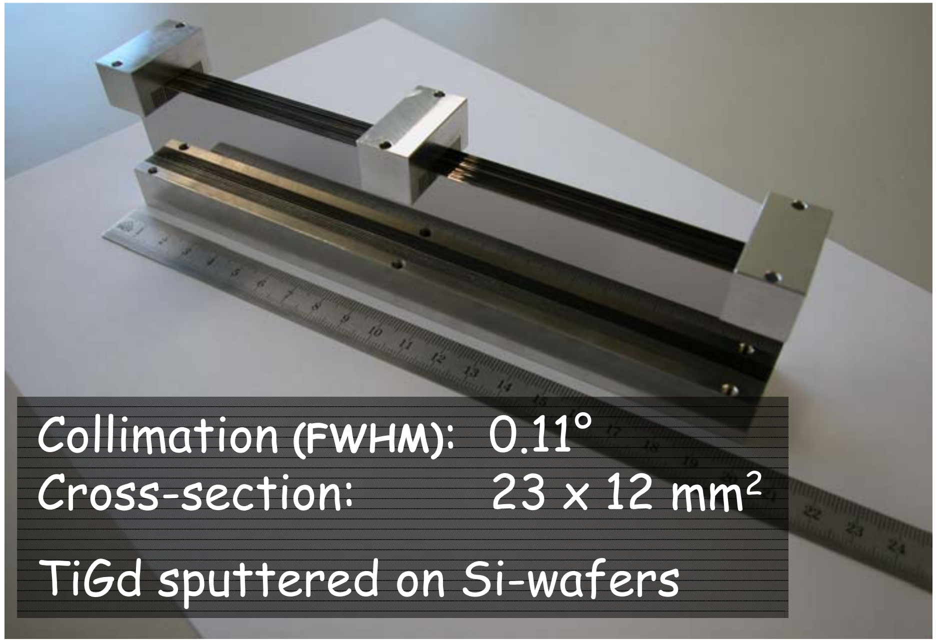
dilution insert

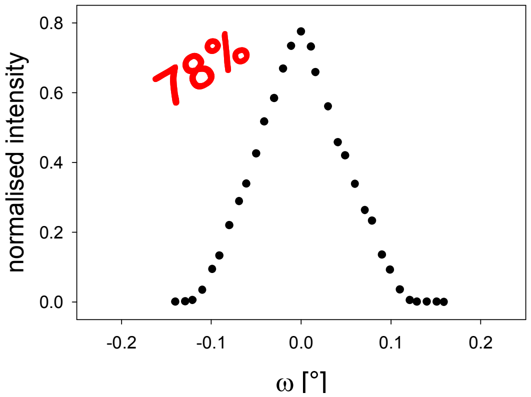


[P. Roubeau, *J. Phys.* **39** (1978) C6-1146]
[B. van den Brandt et al., *Nucl. Instr. Meth.* A **289** (1990) 526]

cryostat details

new neutron collimators





[F.M. Piegsa, *Nucl. Instr. Meth.* A **603** (2009) 401]

Compare (state of the art Si collimators):

T. Krist et al., *Physica B* **356** (2005) 197

Collimation (FWHM): 0.37°
Peak Transmission: 83 %

target materials & nuclei

Since 1971 the PSI polarised target group has realised a large number of polarised target systems for demanding particle physics experiments.

For each polarised target experiment specific R&D is necessary:

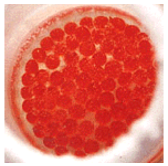
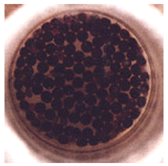
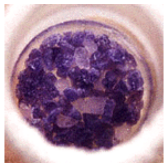
cryogenics, magnets, NMR, particle-beam restrictions, …

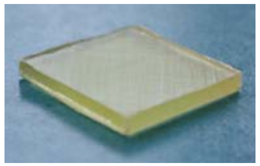
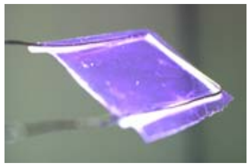
„target material“ containing polarised nuclei

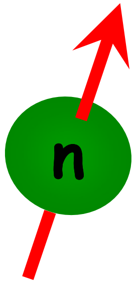
Polarised nuclei: **p**, **d**, ^{6}Li, ^{7}Li, ^{10}B, **^{13}C**, ^{15}N, ^{19}F, ^{27}Al, ^{139}La, …

In frozen alcohols (butanol, propandiol, glycerol, …), plastics (PS, PE, …), Li-hydrates, Li-deuterates, crystals and ammonia.

doped with free radicals: EHBA-Cr(V), TEMPO, irradiated, etc.

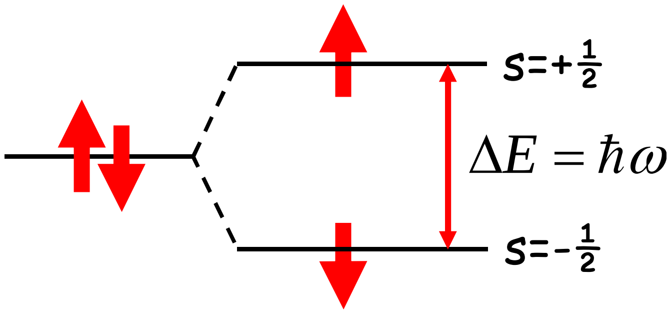






How does the neutron spin interact ?

spin-dependent nuclear interaction	magnetic interaction
$V_{\text{fermi, inc}} \propto b_i \, \vec{s} \cdot \vec{I} \; \delta(\vec{r})$	$V_{\mathbf{magn}} = -\gamma_{\mathbf{n}} \hbar \, \vec{s} \cdot \vec{B}$
↓	↓
pseudomagnetic precession	**magnetic Larmor precession**



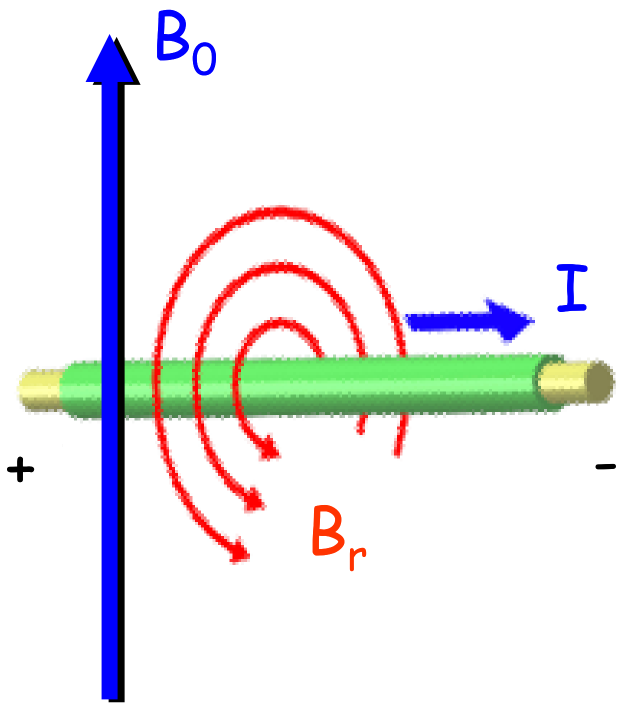
[Abragam & Goldman, *Nuclear Magnetism* (1982) Oxford Univ. Press]

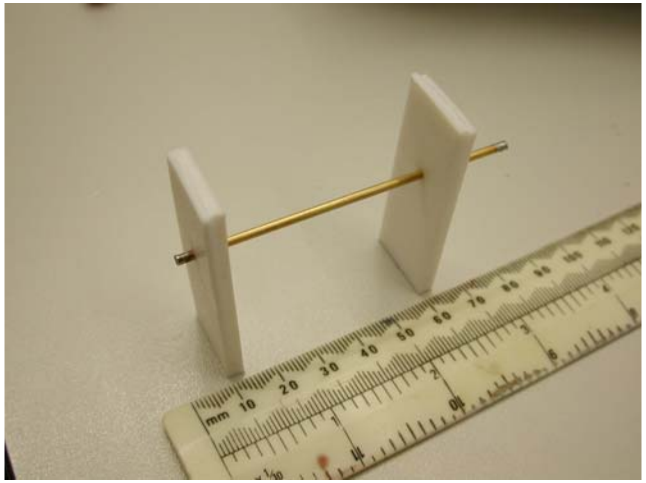
neutron scattering lengths table

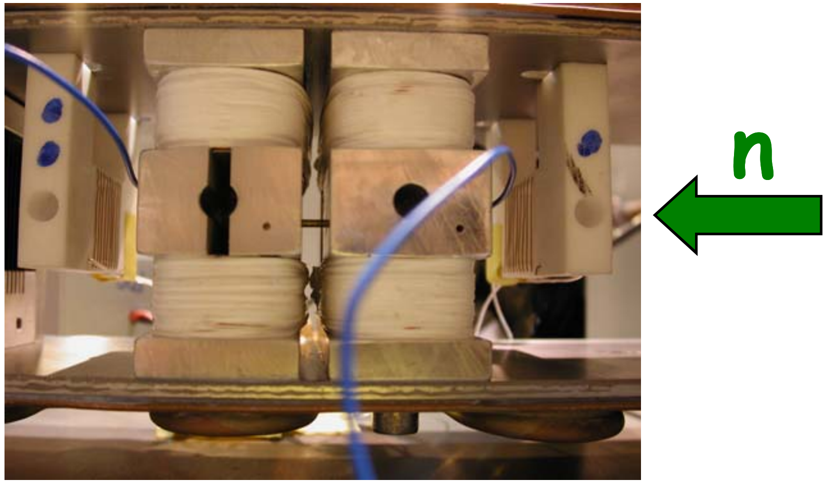
Nucleus	natural abund.	Spin I	b_c [fm]	b_i [fm]	$\gamma/2\pi$ [MHz/T]
^{1}H Proton	**99.985 %**	**1/2**	**-3.742 (1)**	**25.274(9) ***	**42.576**
^{2}H Deuteron	0.015 %	1	6.671 (4)	4.04(3) *	6.536
^{3}He	0.00014 %	1/2	5.74 (7)	-2.37(2) **	32.43
^{14}N	99.63 %	1	9.37 (2)	2.0(2) *	3.076
^{27}Al	100 %	5/2	3.449 (5)	0.26(1) *	11.09
^{51}V	99.75 %	7/2	-0.402 (2)	6.35(4) *	11.19

* [V.F. Sears, Neutron News **3** (1992) 27]
** [O. Zimmer et al., EPJ dir. A **1** (2002) 1]

- **Protons cause reasonable spin precession due to large b_i and γ.**
- **Imaging of polymers and soft-matter should be possible.**

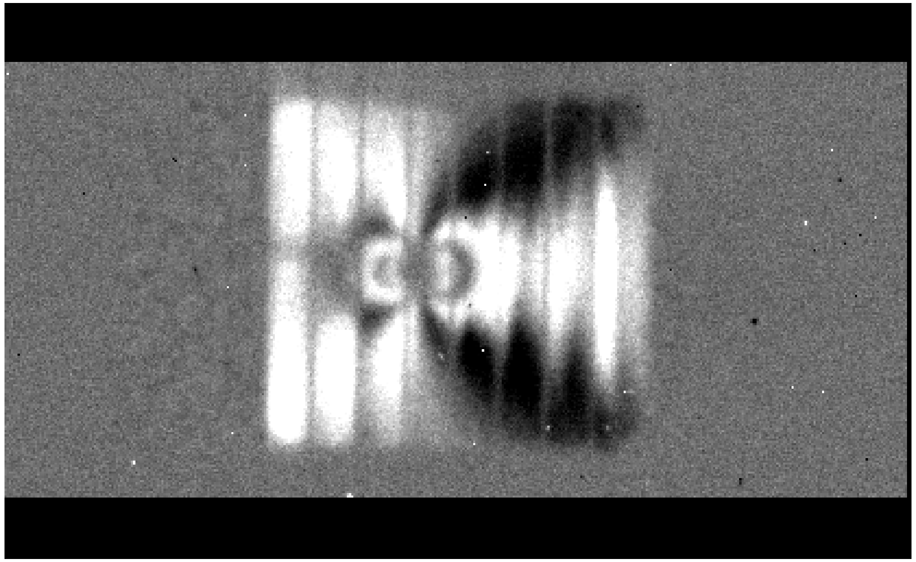






$B_0 \gg B_r$ → only sensetive to $B_{r,z} // B_0$

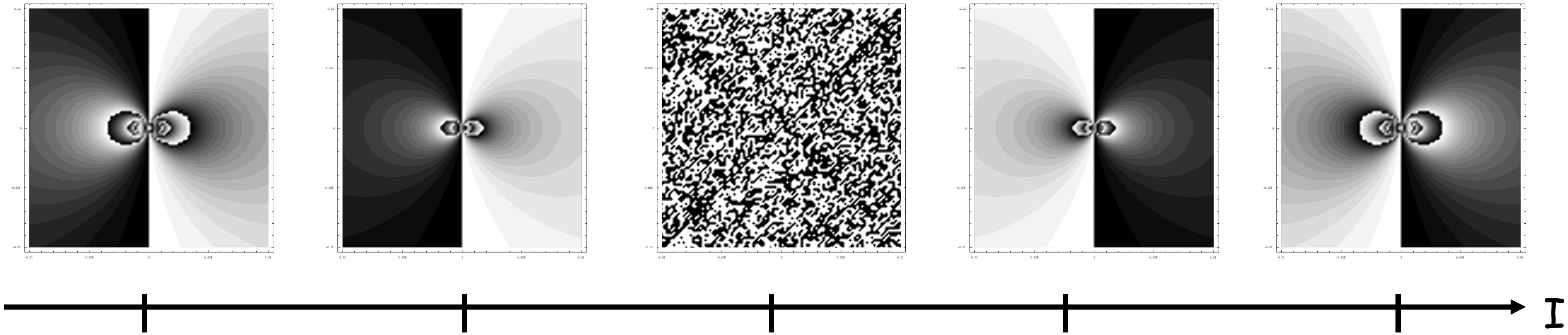
longitudinal wire



Currentscan: from -5 … +5 A

on one frequency

Qualitative Simulation:



Polarised nuclei for NMR & MRI

^{13}C, ^{6}Li, ^{15}N

B. van den Brandt, P. Hautle, J.A. Konter, F. Kurdzesau

J. van der Klink, A. Comment, F. Kurdzesau, S. Jannin, W.Th. Wenckebach & R. Grütter (CIMB Lausanne)

F. Piegsa – June 9th 2009 – St. Petersburg

motivation for DNP-enhanced NMR/MRI

^{13}C-labeling is widely used to mark **molecules of metabolic interest** in NMR/MRI, e.g. to investigate brain metabolism *in vivo.*

Limitation: ^{13}C has a low sensitivity ($P_{^{13}C}$ ~ **0.0008 %** @ 300 K, 9.4 T)	⟷	**Dynamic Nuclear Polarisation** ($P_{^{13}C}$ ~ **10 %** @ 1.2 K, 3.5 T)

one can dissolve a frozen polarised sample

transfer the polarised liquid out of the cryostat to a NMR/MRI-setup

and it is still polarised when injected (in the animal)

[J. Wolber et al., *NIM A* **526** (2004) 173]

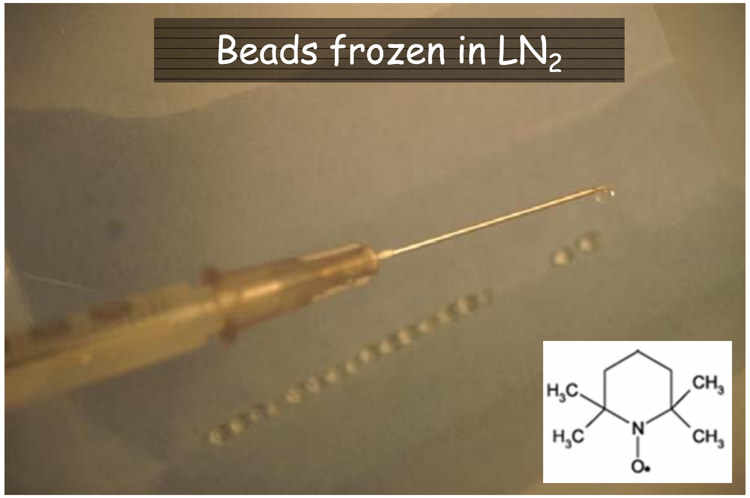
polarisation setup at EPF Lausanne

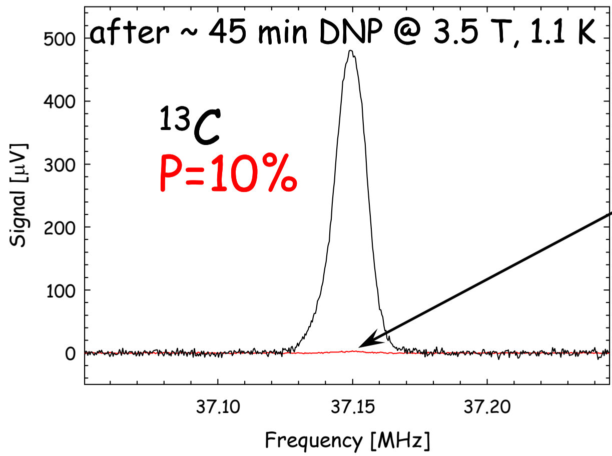
Setup:

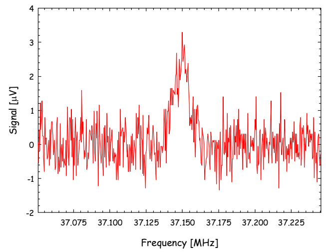
- Cryostat fits in a standard wide-bore NMR magnet **3.35 T** (Ø = 88 mm)
- Base temperature ~ **1.1 K** with low L^4He consumption (~ 60 l/week)
- 94 GHz microwave source ~ 50 mW
- Fast sample loading and polarisation in about one hour

Extensive tests of sample compounds and concentrations @ PSI:

e.g. 3M Sodium Acetate (^{13}C labeled) embedded in a polarised matrix of D_2O/d-ethanol doped with $2x10^{19}$/ml TEMPO.







no DNP
P = 0.08%

Method generally applicable to polarise nuclei in molecules.

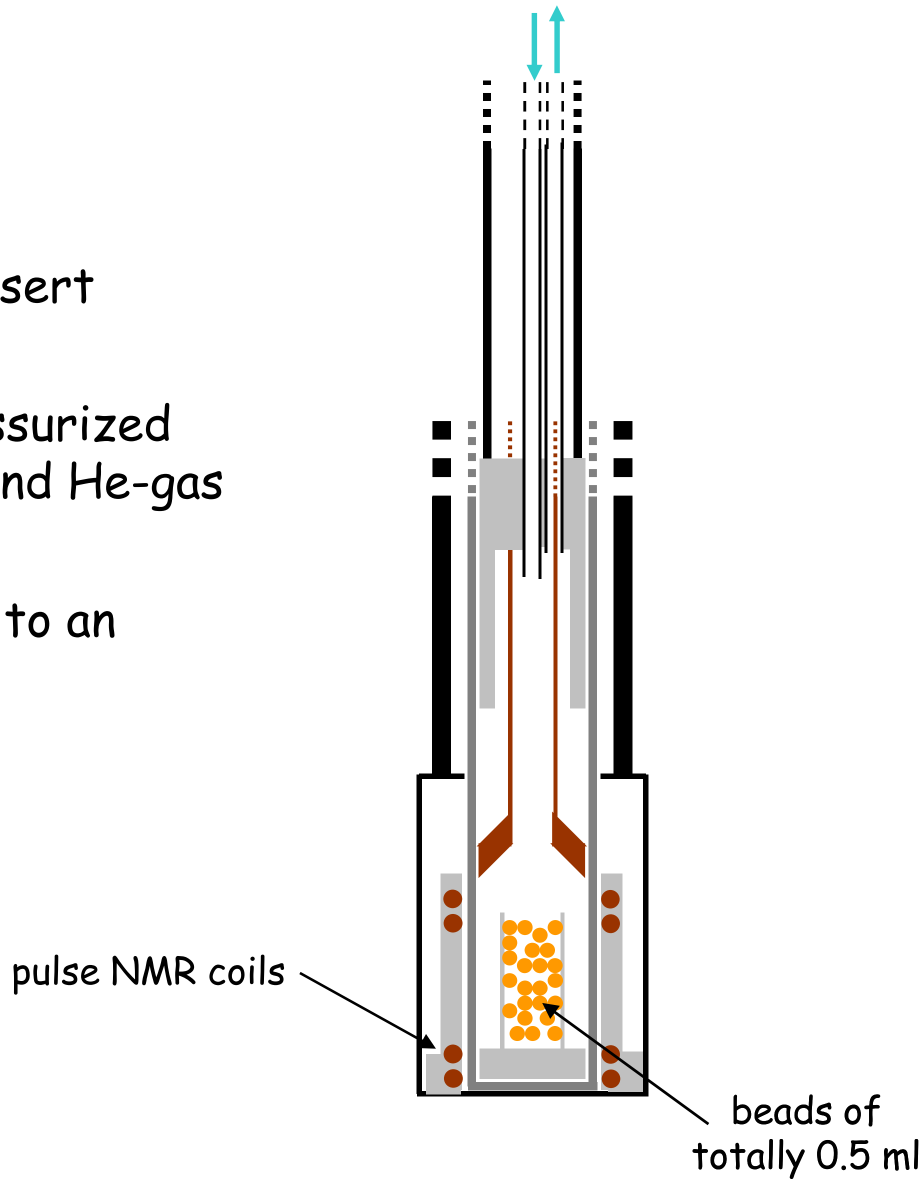
And also other species: ^{6}Li, ^{15}N, ^{31}P, …

[*J. Phys. D: Appl. Phys.* 41 (2008) 155506]

dissolution - procedure

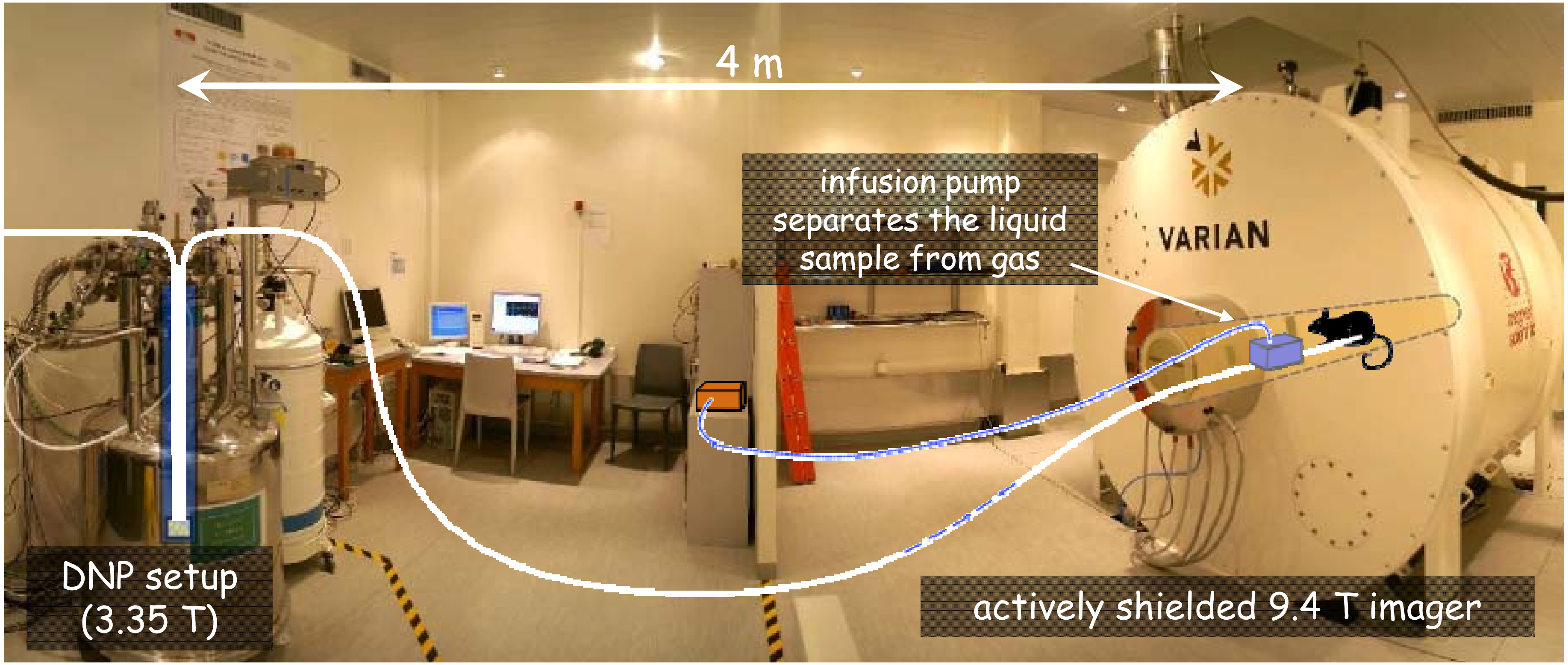
- DNP-insert replaced by dissolution-insert
- Dissolution by injection of highly pressurized water (several bars at about 150°C) and He-gas
- Dissolution outlet directly connected to an infusion device

[*Concepts Magn. Resonance* **31B** (2007) 255]



Principle follows:
[Ardenkjær-Larsen et al.,*PNAS* **100** (2003) 10158]

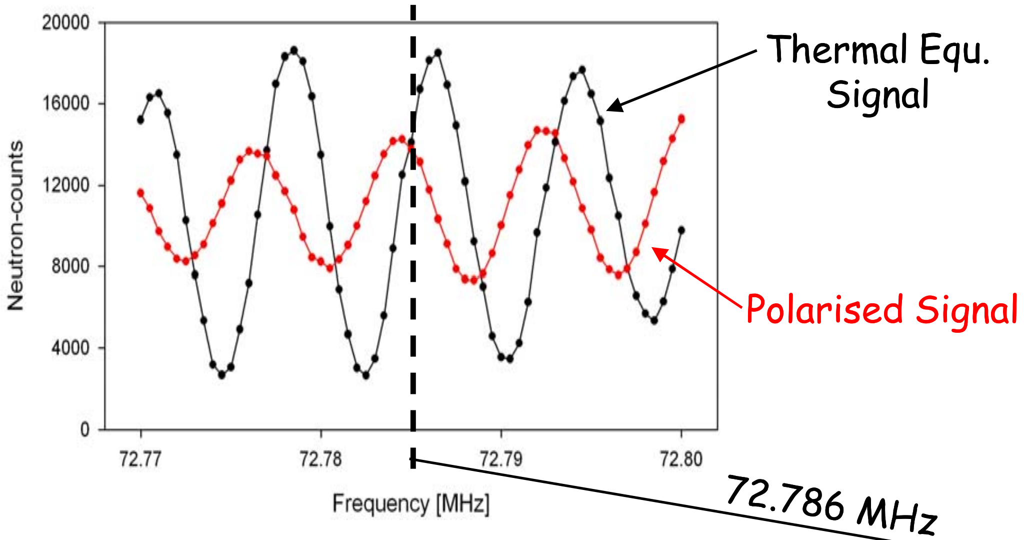
dissolution and sample transfer

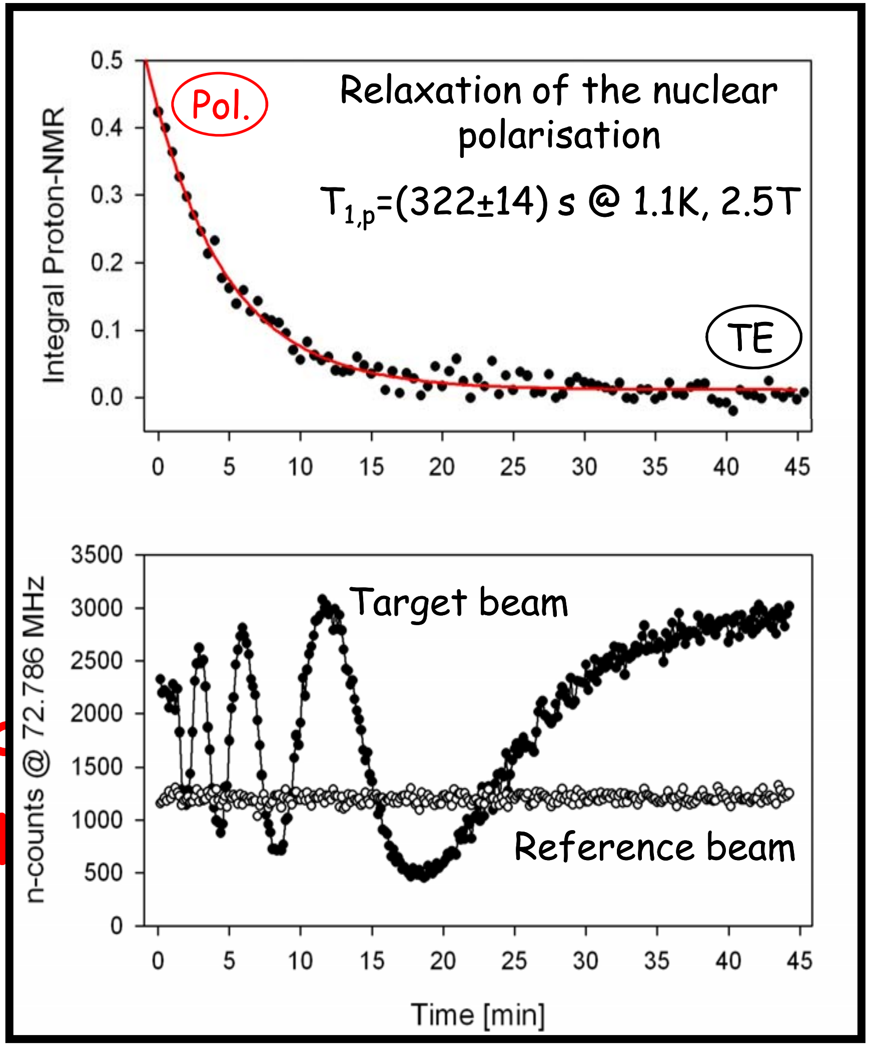


- Dissolution, transfer and infusion takes about 15 sec (loss ~ 50%)
- Remaining Enhancement-factor ~ 3000
- DNP and MRI at different magnetic fields

pseudomagn. phase-shift due to DNP

<u>Sample:</u> d-PS (ARMAR: 98%D) doped with: 2.7×10^{19} d-TEMPO/ml
thickness = 1.6 mm - 16.12.2005





Measuring time: <u>45 min</u> each !

How b

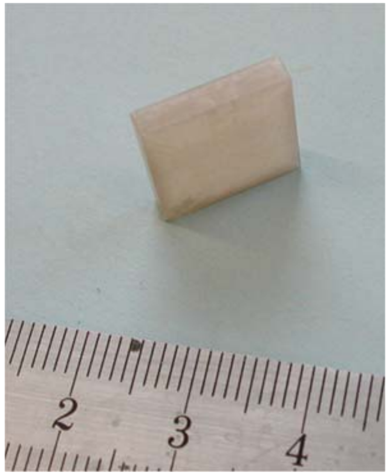
ap

$P_p = 17\%$ $P_d = 12\%$ (from NMR)

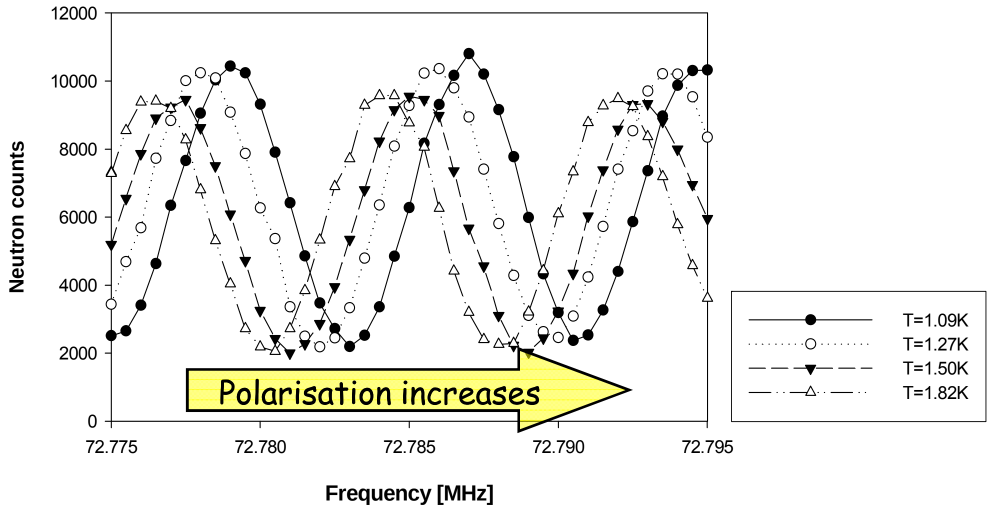
$\varphi^*_{expect.} = (1503 \pm 132)^\circ$

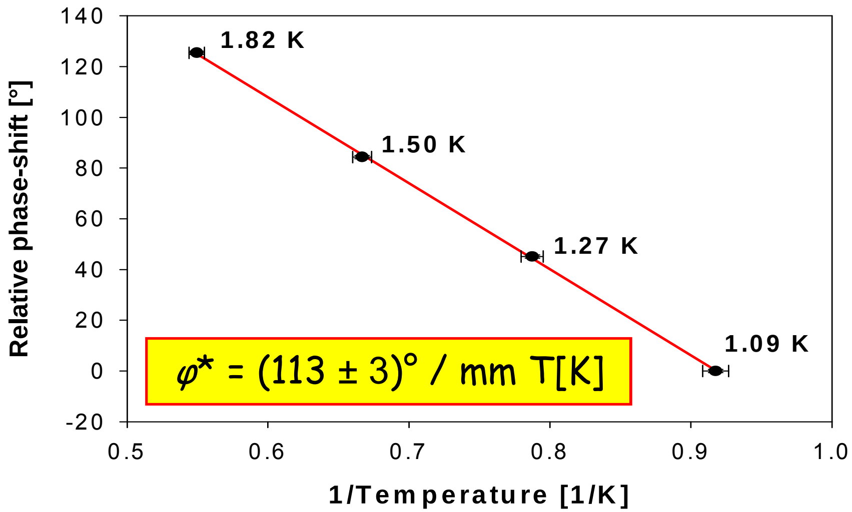
$\varphi^*_{meas.} = (1350.2 \pm 1.5)^\circ$ → 10^{-3}

example for pseudomagnetic precession



- 3 mm thick n-Polystyrene measured at 2.5 Tesla and various temperatures (25.11.05)
- Thermal equilibrium polarisation of proton spins cause pseudomagnetic precession





describing nuclear forces at low energies

Potential models for nuclei have a limited predictive power and need an enormous amount of input parameters.

Instead: **Effective field theories (EFT's)** provide systematic and model independent descriptions. They use point like & effective couplings (like Fermi's theory).

[Bedaque et al., Nucl. Phys. A **714** (2003) 589]
[Griesshammer, Nucl. Phys. A **744** (2004) 192]

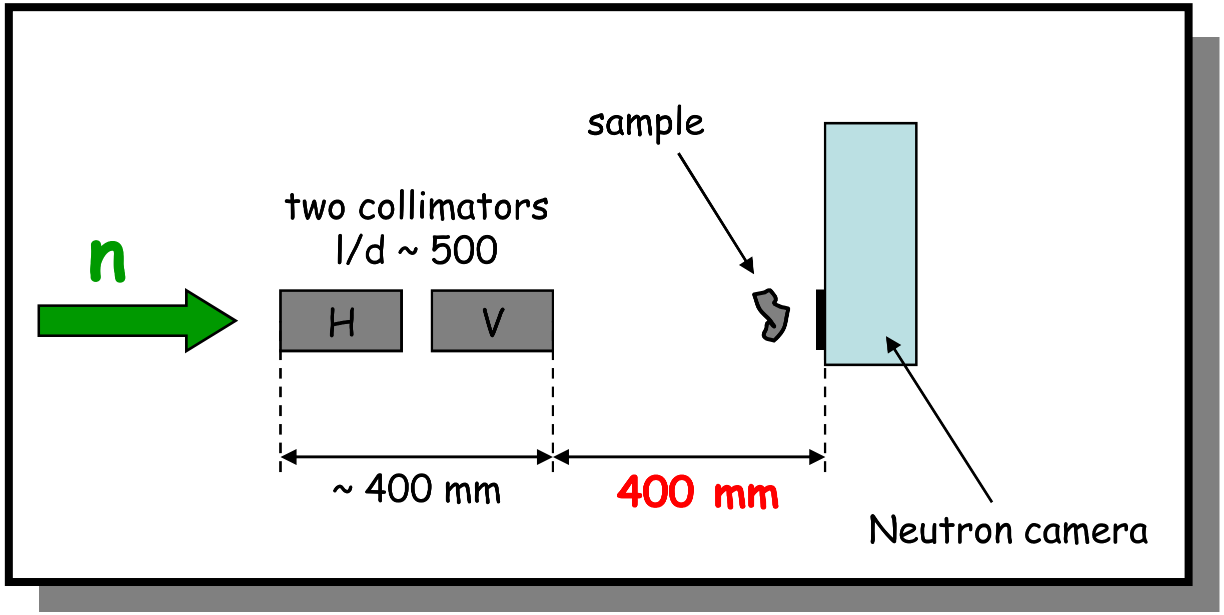
To make predictions on the NNLO-level, EFT's need only two independent experimental input parameters (LEC's) of the the 3-nucleon system (e.g. for nuclear synthesis / big-bang reactions etc.):

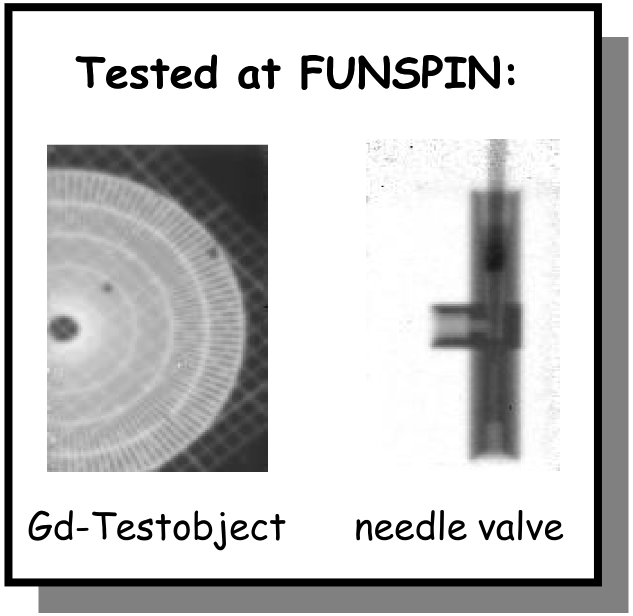
1. Triton binding energy ($5\mathrm{x}10^{-7}$)
2. **Doublet scattering length $b_{2,d}$ of the nd-system (6%)**

$$b_{2,d} = b_{c,d} - \sqrt{2}\, b_{i,d}$$

[Schoen et al., Phys Rev. C **67** (2003) 044005]

"short-range" neutron imaging





collimation × closer to beam-exit × larger beam =

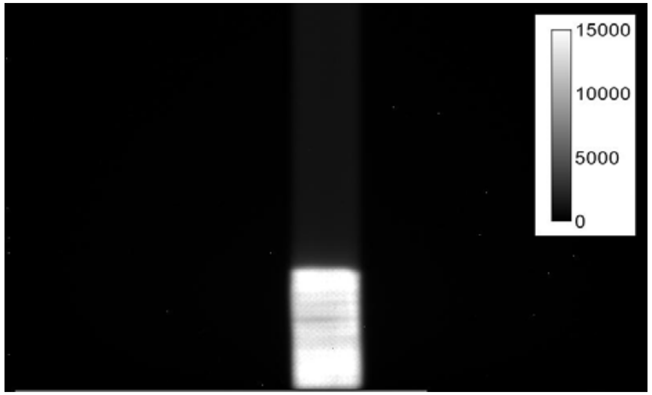
= 0.01 × 20^2 × 1… = 4…

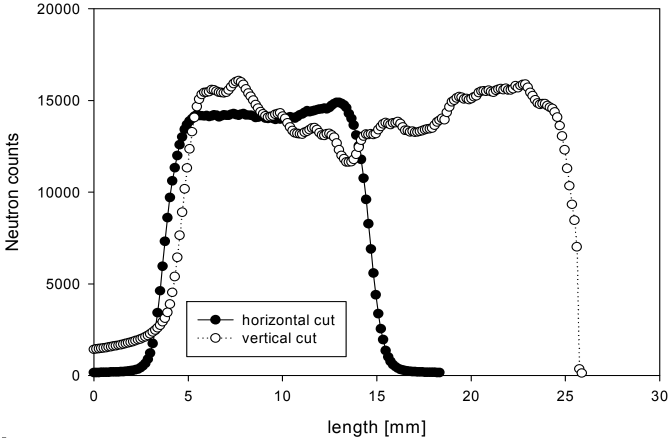
Neutron flux density gain compared to standard pin-hole geometry is larger than 4.

Gd-Testobject: [C. Gruenzweig et al., *Rev. Sci. Inst.* **78** (2007) 053708]

FUNSPIN beam-image 2.7.2008 (phi=+1.045°, h=11.26 mm, exp. time=60 sec)

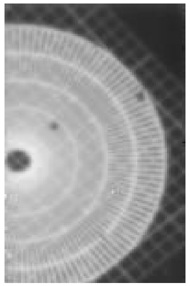
Distance = 0.4 m



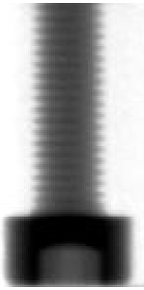


$FWHM_H$ = 10.8 mm (before: 8.6 mm)
$FWHM_V$ = 21.1 mm (before: 20.3 mm)

Test-Imaging in 0.4 m distance:

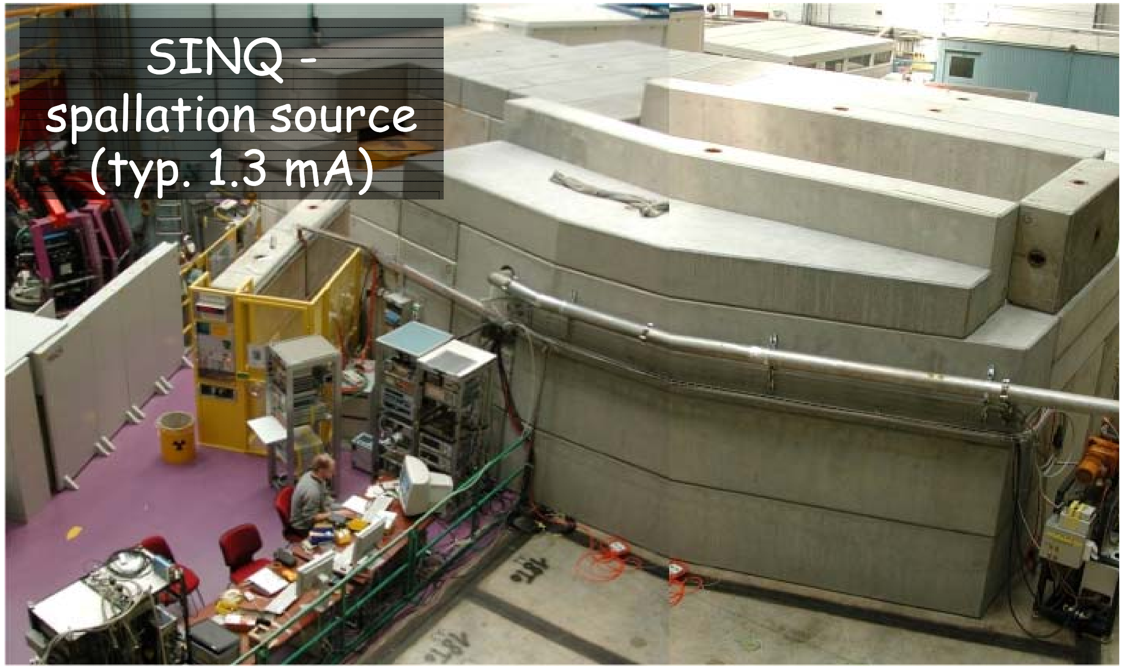

Gd-Testobject

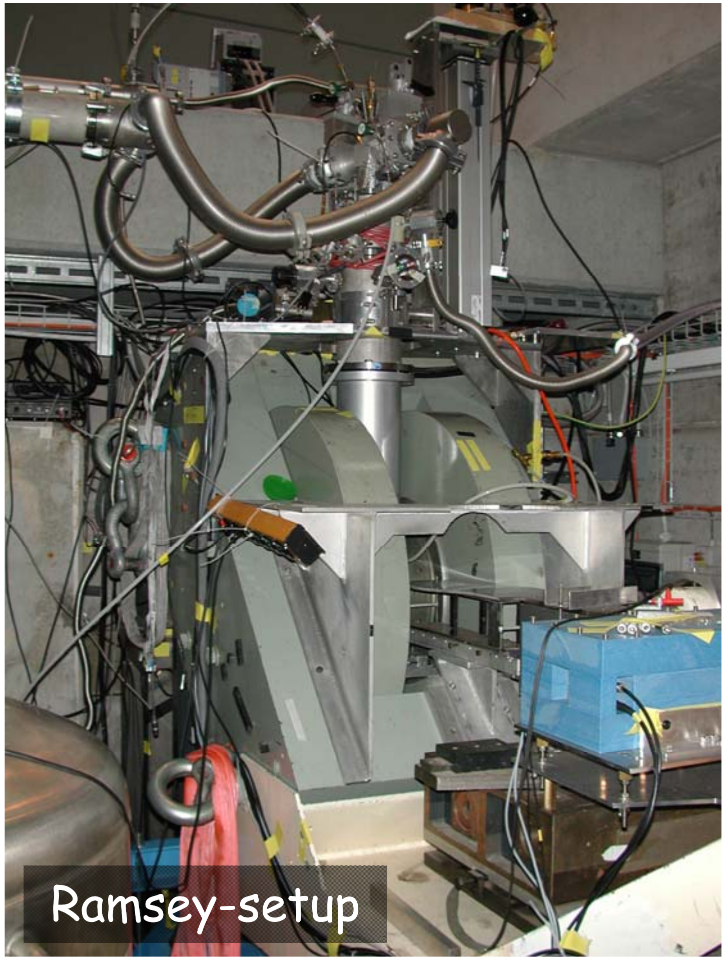

coin

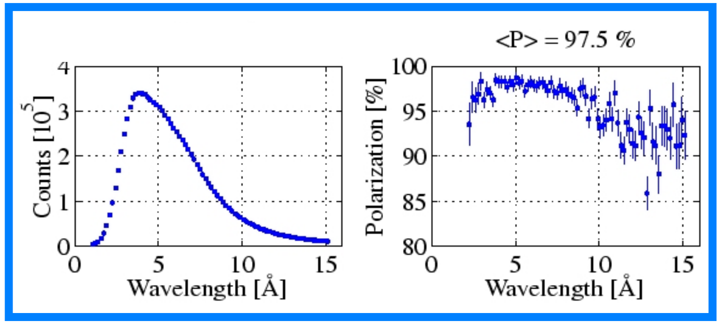

M5-screw


needle valve

FUNSPIN beam line at PSI



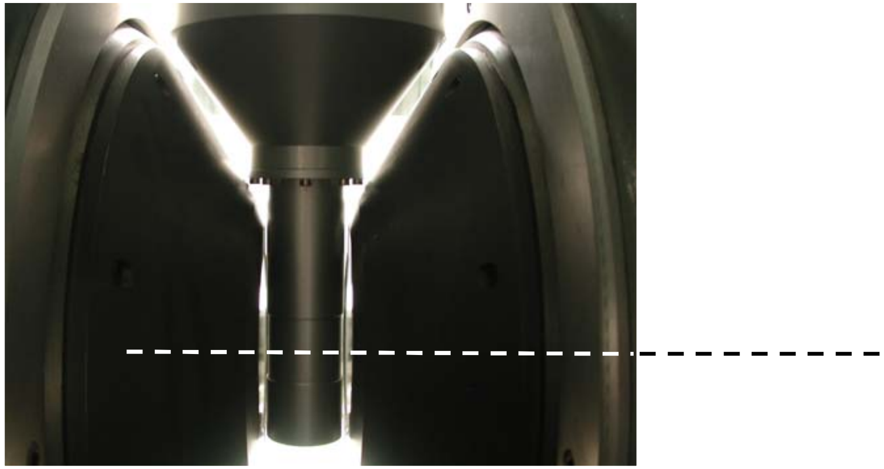


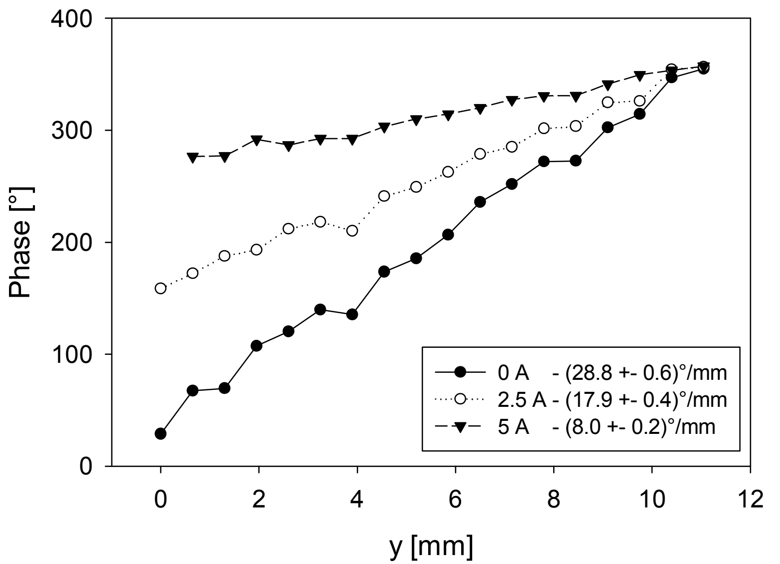


wavelength-integrated neutron-flux density:
$\Phi = 2.46 \times 10^8$ [n/cm²·s·mA] - **polarised**

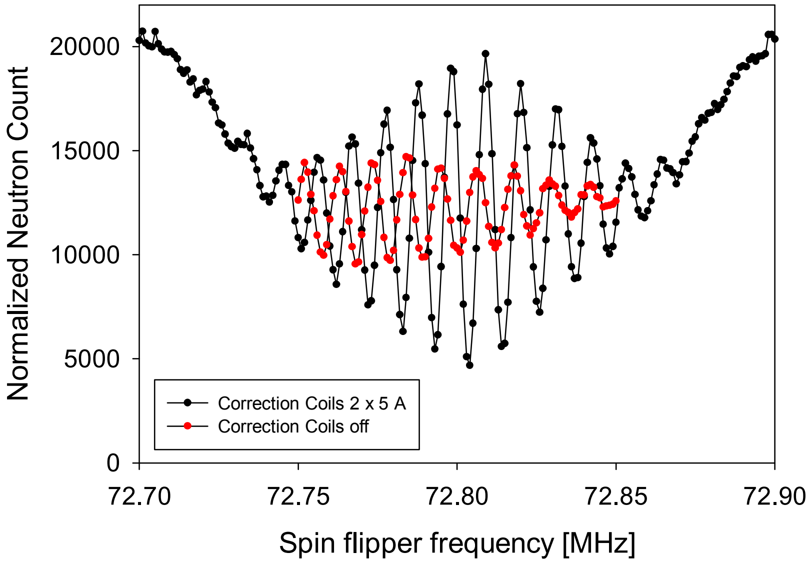
[J. Zejma et al., NIM A 539 (2005) 622]

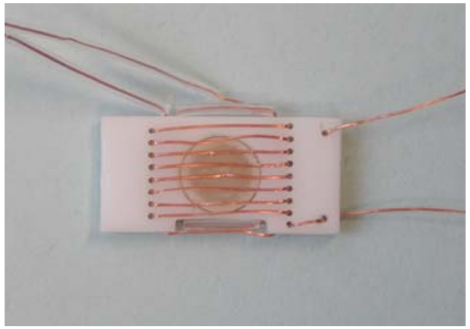
large target beam (7x8 mm²)

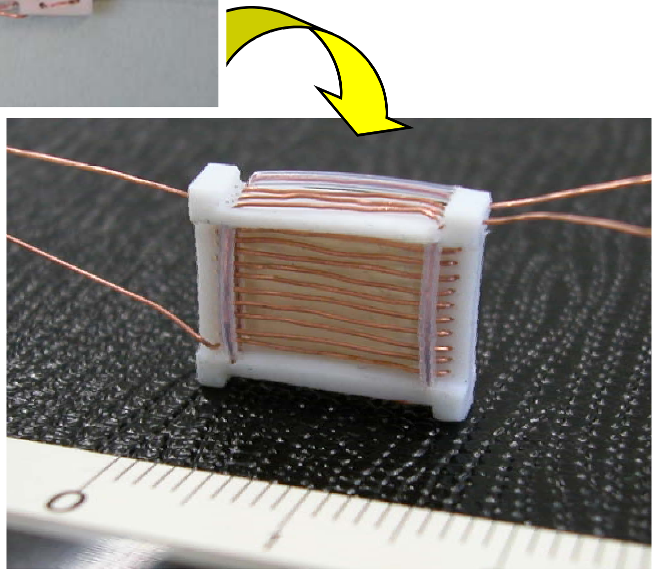




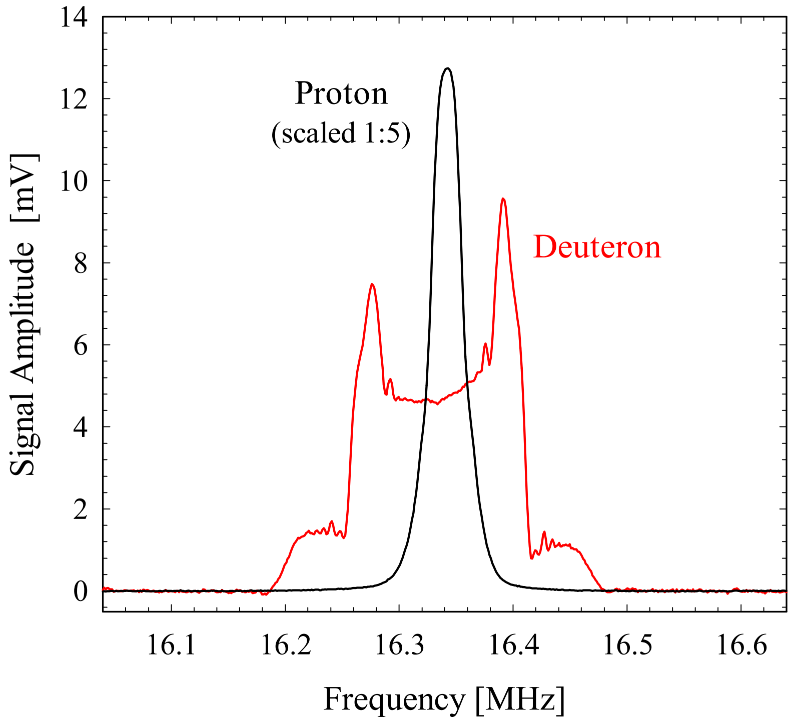
Horizontal magnet homogeneity
with correction coils at 5 A: 2 x 10^{-4}/cm



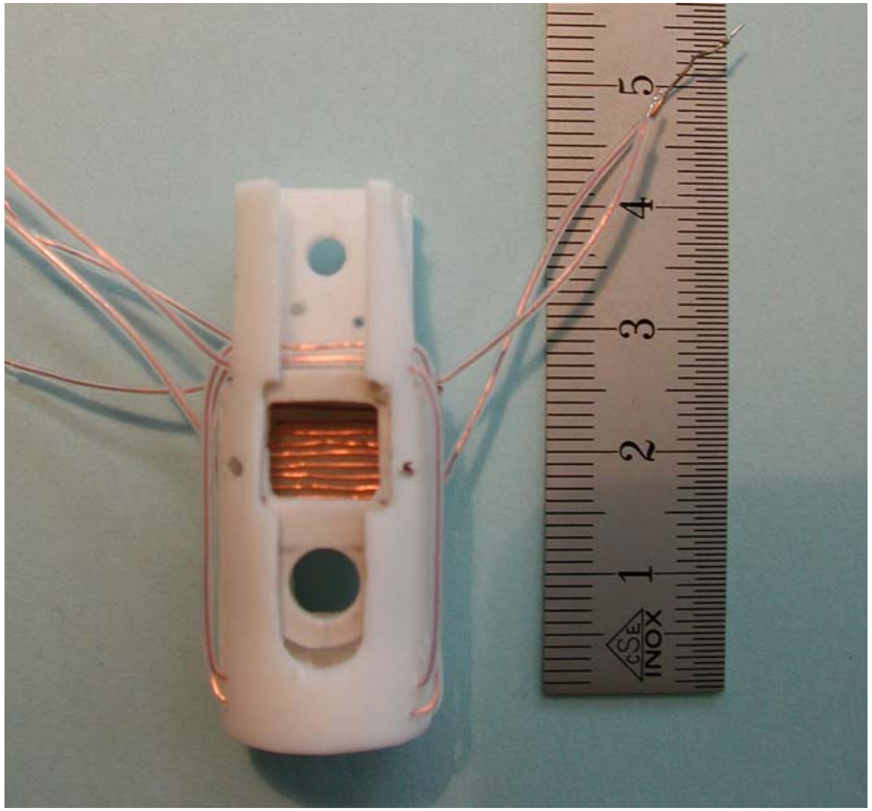


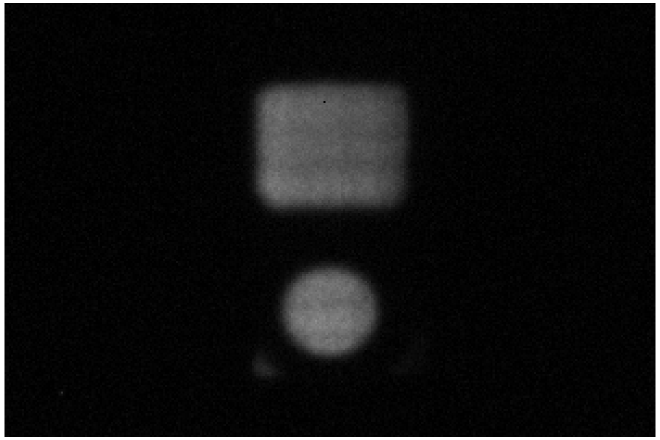


New sample: 10 x 8 x 1.3 mm^3 (110 mg)



NMR signals could be improved by a factor of 4, due to larger sample and better coil design.



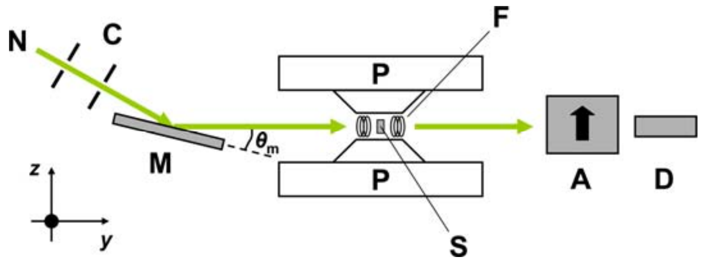


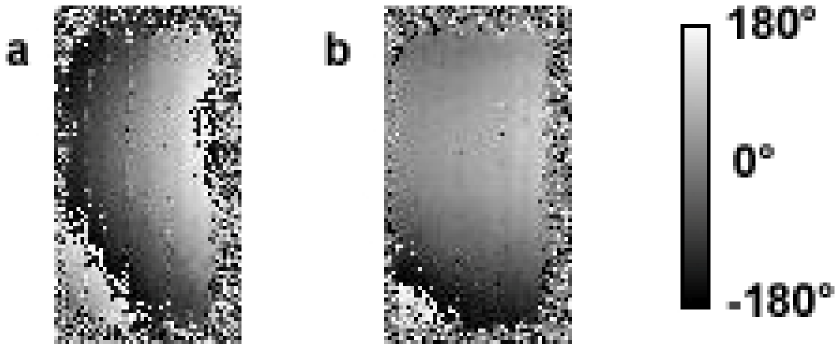
Movie of the two neutron beams
during cryostat cooldown

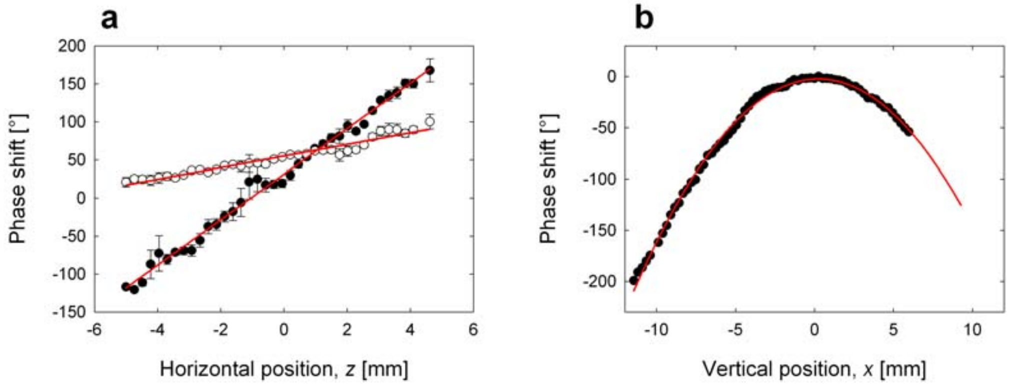
24 hours, 300K to 4K – shrinkage of 1.5 mm

(25. - 26.08.2008)

The new collimation system even allows to perform neutron radiography.


N
C
F
P
M
P
A
D
S
z
y


a
b
180°
0°
-180°


a
Phase shift [°]
Horizontal position, z [mm]
b
Phase shift [°]
Vertical position, x [mm]