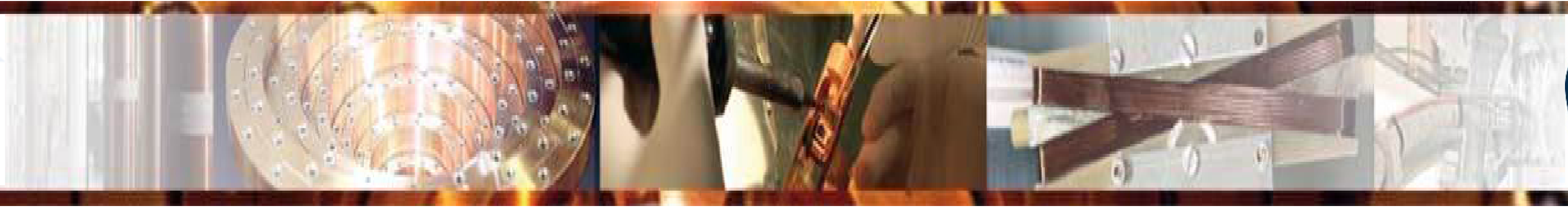


LNCMI

cnrs dépasser les frontières

IMMW21

International Magnetic Measurement Workshop

24th – 28th June 2019

High precision magnetic measurements at very high magnetic fields up to 37 T

Kevin Paillot and Steffen Krämer

LNCMI Grenoble

June, 24th 2019

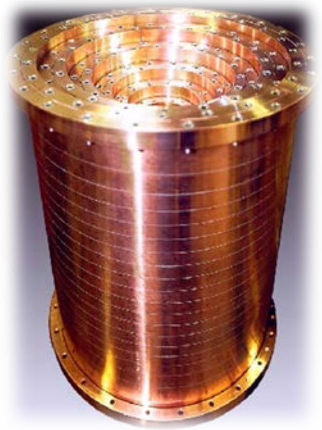
UNIVERSITÉ Grenoble Alpes

INSA INSTITUT NATIONAL DES SCIENCES APPLIQUÉES TOULOUSE

UNIVERSITÉ TOULOUSE III PAUL SABATIER Université de Toulouse

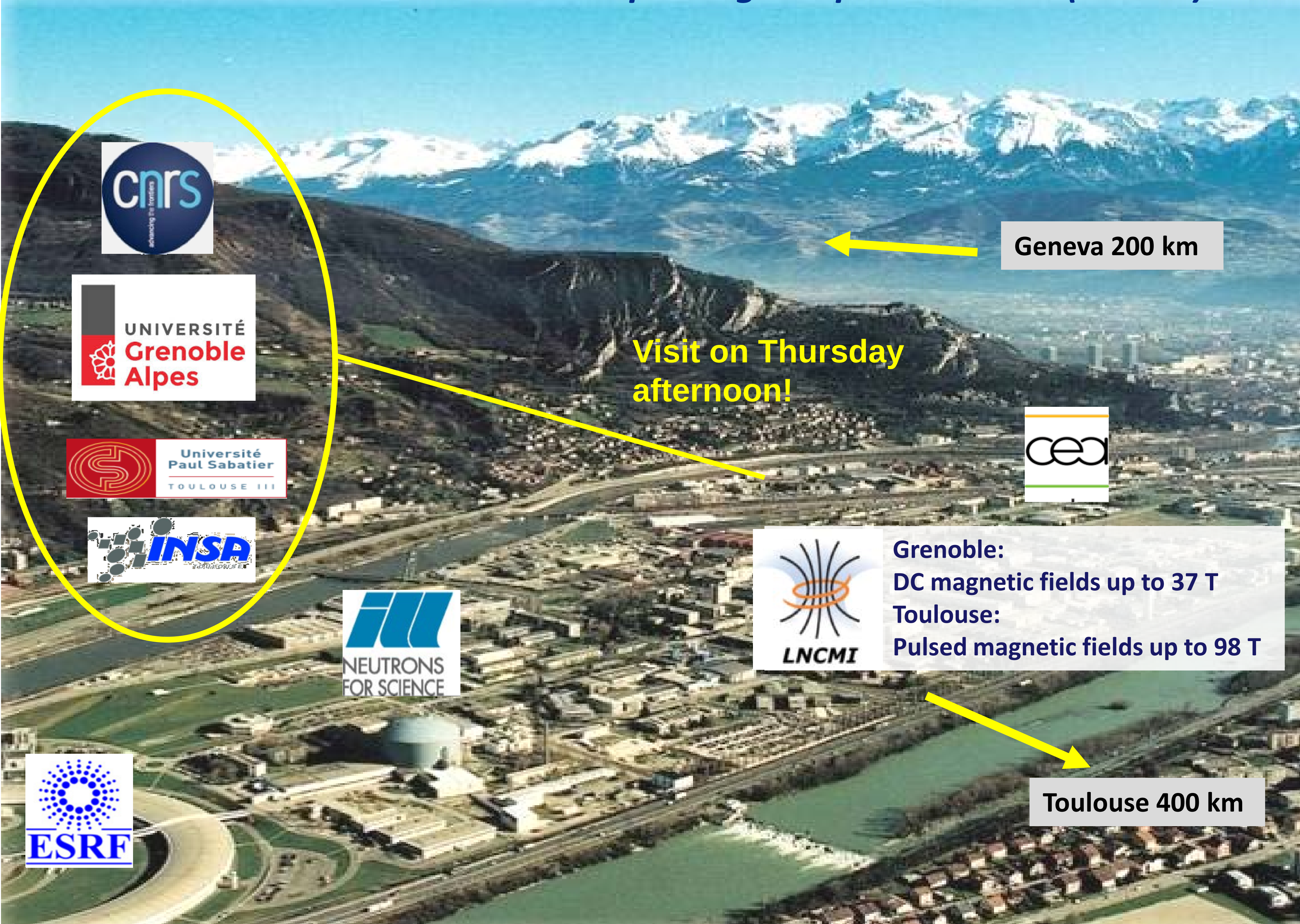
EMFL European Magnetic Field Laboratory

Magnetic measurements at LNCMI - collaborators



- **24 MW power converter team:**
  R. Barbier, C. Grandclément, R. Jaymond, K. Juge, B. Vincent
- **DC magnet team:**
  C. Auternaud, F. Debray, J.-L. DeMarinis (retired), O. Jay,
  M. Kamke, M. Pelloux, R. Raison, P. Sala(retired), C. Trophime,
  E. Vernay
- **Instrumentation team:**
  R. Pankow, E. Yildiz
- **NMR team:**
  M. Horvatić
- **Mechanical workshop:**
  T. Disparti, C. Mollard, D. Ponton, J. Spitznagel, J.-M. Tudela,

Laboratoire National des Champs Magnétiques Intenses (LNCMI)



Geneva 200 km

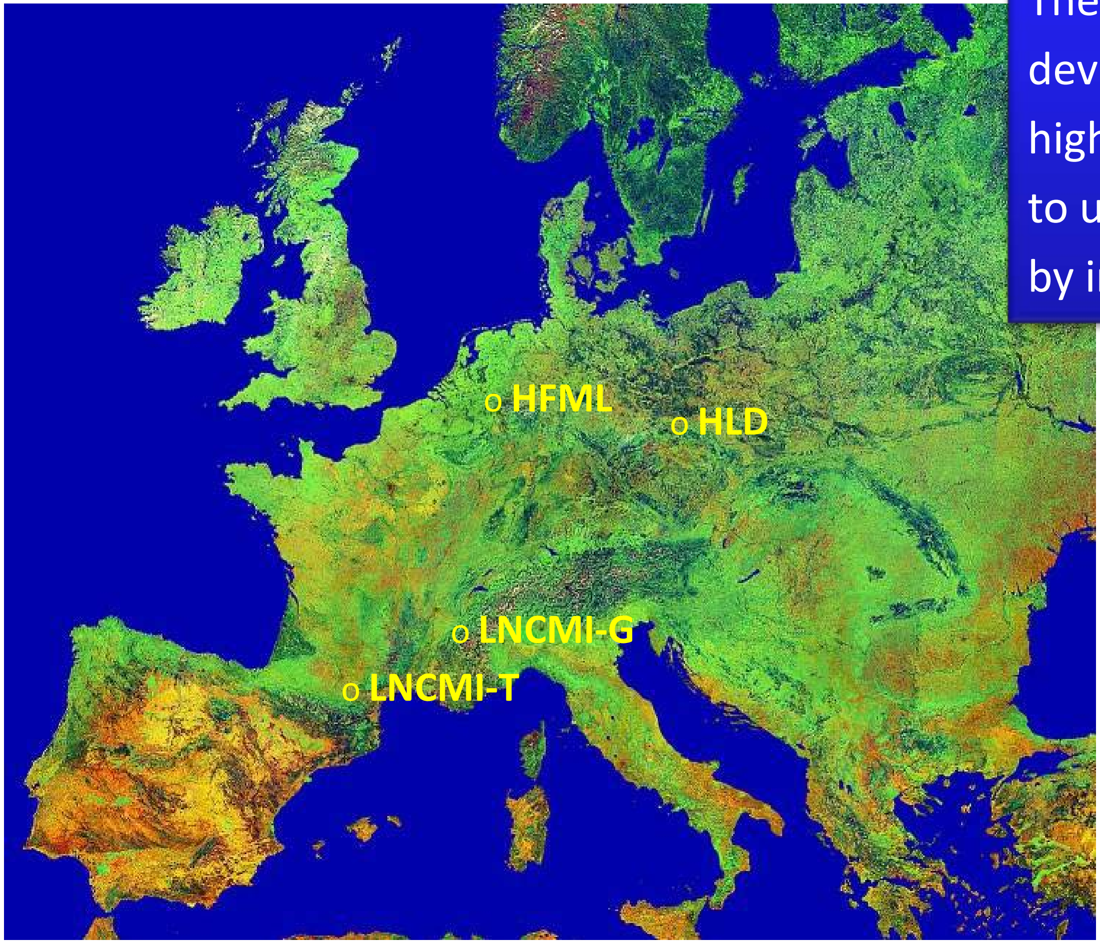
Visit on Thursday afternoon!

Grenoble:
DC magnetic fields up to 37 T
Toulouse:
Pulsed magnetic fields up to 98 T

Toulouse 400 km

EMFL – European Magnetic Field Laboratory

European Magnetic Field Laboratory



The EMFL
develops and operates world class
high magnetic field facilities,
to use them for excellent research
by in-house and external users.

EMFL Laboratories:

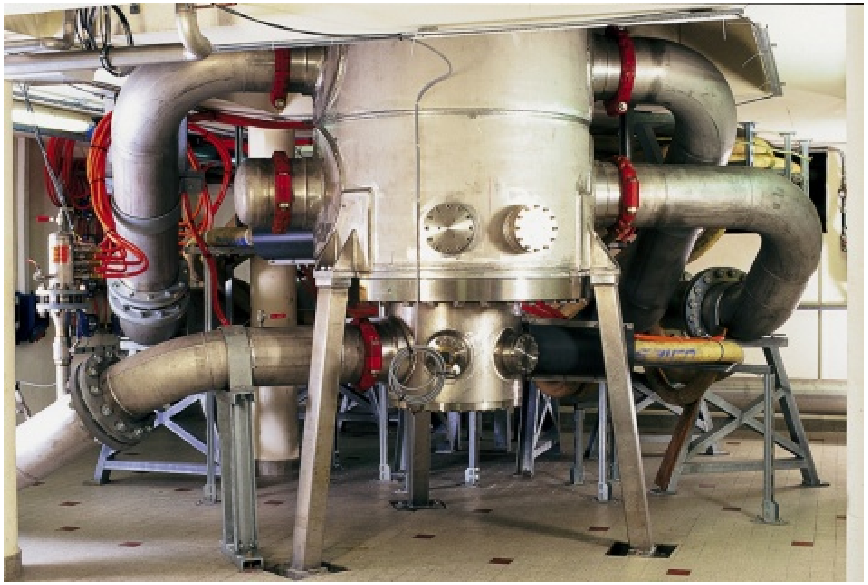
- HFML Nijmegen, NL
- HLD Dresden, D
- LNCMI Grenoble/Toulouse, F

Further EMFL members:

- United Kingdom
- Poland

Information and common platform for user projects: http://www.emfl.eu

LNCMI Grenoble: Mission and In-house science

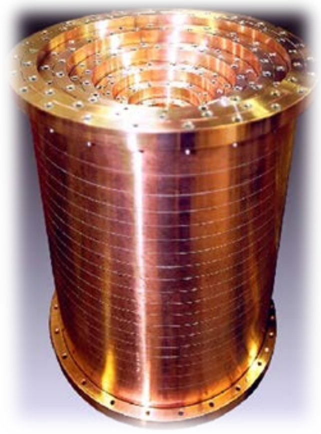


- **Development**
  of high magnetic field installations and instrumentation
- **In-house research**
  in high magnetic fields
- **Access**
  to all qualified French
  and European high field users

In-house science

- Correlated electrons : High T_c superconductors, heavy fermions,
- Organic conductors
- Quantum magnetism
- Semiconductors, graphene, graphite
- Molecular magnetism; synthesis, characterization, EPR
- Advanced magneto-optics
- Applied superconductivity: materials, conductors, devices
- Magneto-science: levitation, elaboration under magnetic field

Magnetometry at LNCMI



- **Science in high magnetic fields:**
  - *Metrology – precise value of B*

    NMR: precise knowledge of B is mandatory for precise NMR
  - *Spatial field distribution – precise distribution of B*

    NMR: resolution

    Magnetization: magnetic torque, magnetic force

    Levitation: magnetic force
  - *Time variation of magnetic field – drift and spectrum of B(t)*

    NMR: resolution

    All other techniques: noise

- **Development of high field magnets and power installation:**
  - *Safety and centering of magnets*

    Forces

    Optimal field profiles
  - *Validation of field calculations:*

    Homogeneity

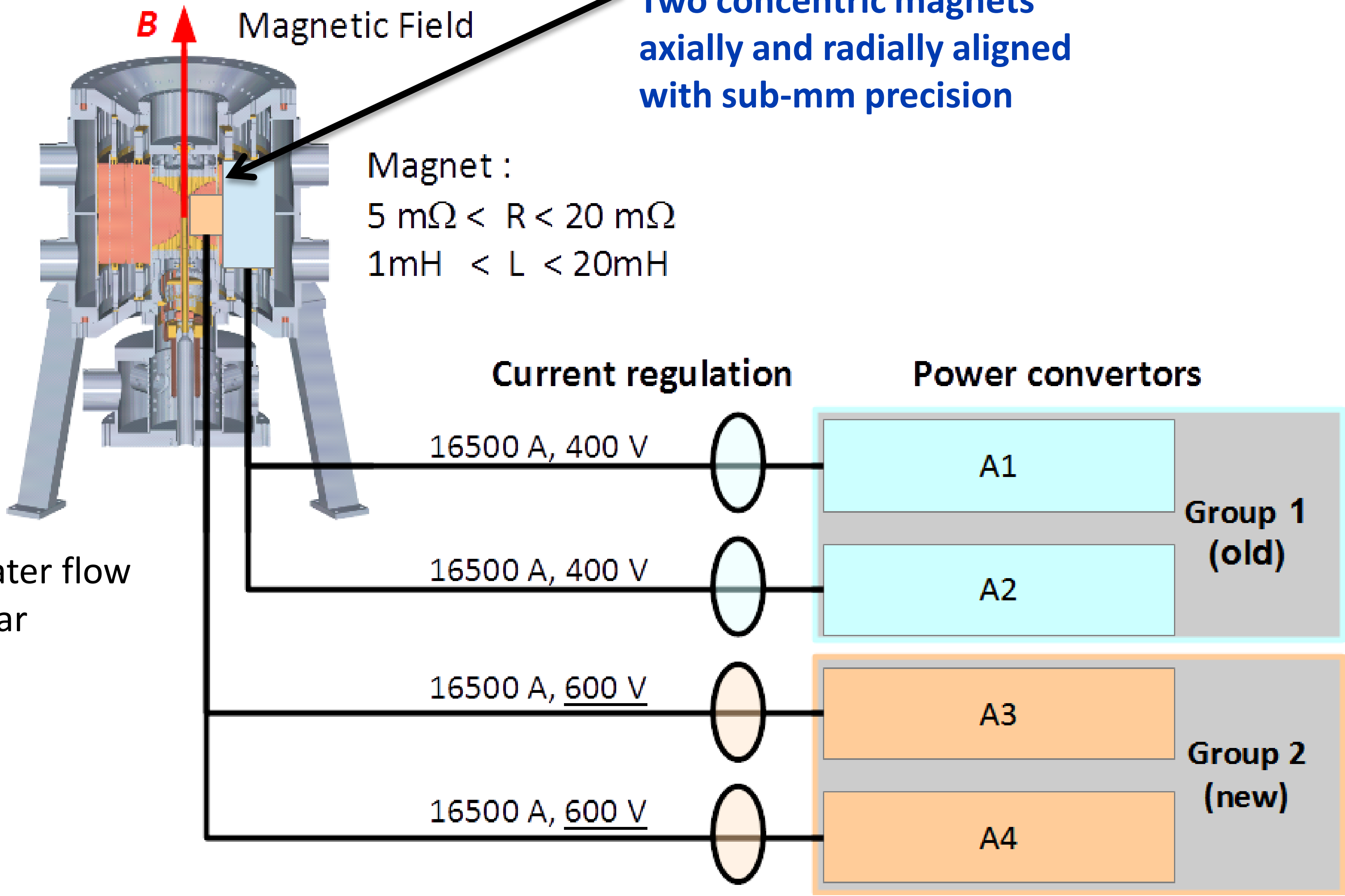
    Levitation zones
  - *Improvement of field quality*

    Spatial resolution

    Temporal stability

Architecture of LNCMI high field facility

example of LNCMI M10 magnet



B Magnetic Field

Two concentric magnets axially and radially aligned with sub-mm precision

Magnet :
5 mΩ < R < 20 mΩ
1mH < L < 20mH

Current regulation

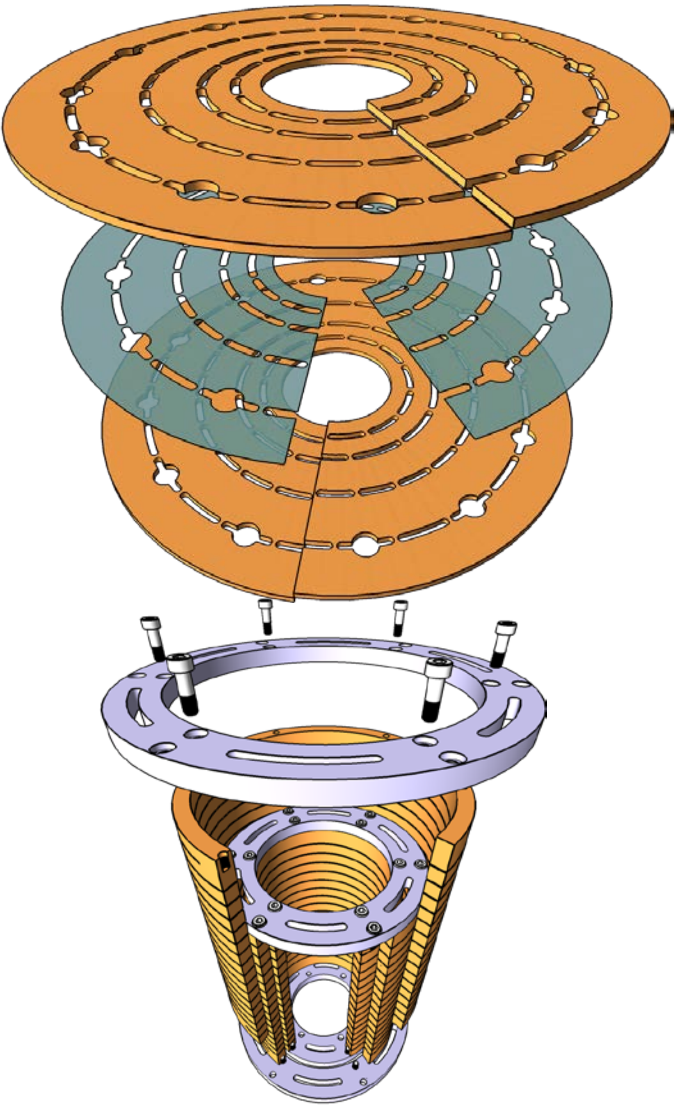
Power convertors

16500 A, 400 V — A1
16500 A, 400 V — A2
Group 1 (old)

16500 A, 600 V — A3
16500 A, 600 V — A4
Group 2 (new)

350 l/s water flow
$\Delta P = 24$ bar
$\Delta T = 30$ K

Generation of ultra-high magnetic field - challenges



Above 23.5 T:
No commercial superconducting magnets (yet).

Resistive magnet: Put current through a coil

$$B \propto I$$

Problem 1: Heating

$$P \propto I^2 \propto B^2$$

Problem 2: Forces

$$\vec{F} \propto \vec{I} \times \vec{B} \Rightarrow F \propto B^2$$

Polyhelix and Bitter solenoids at LNCMI:

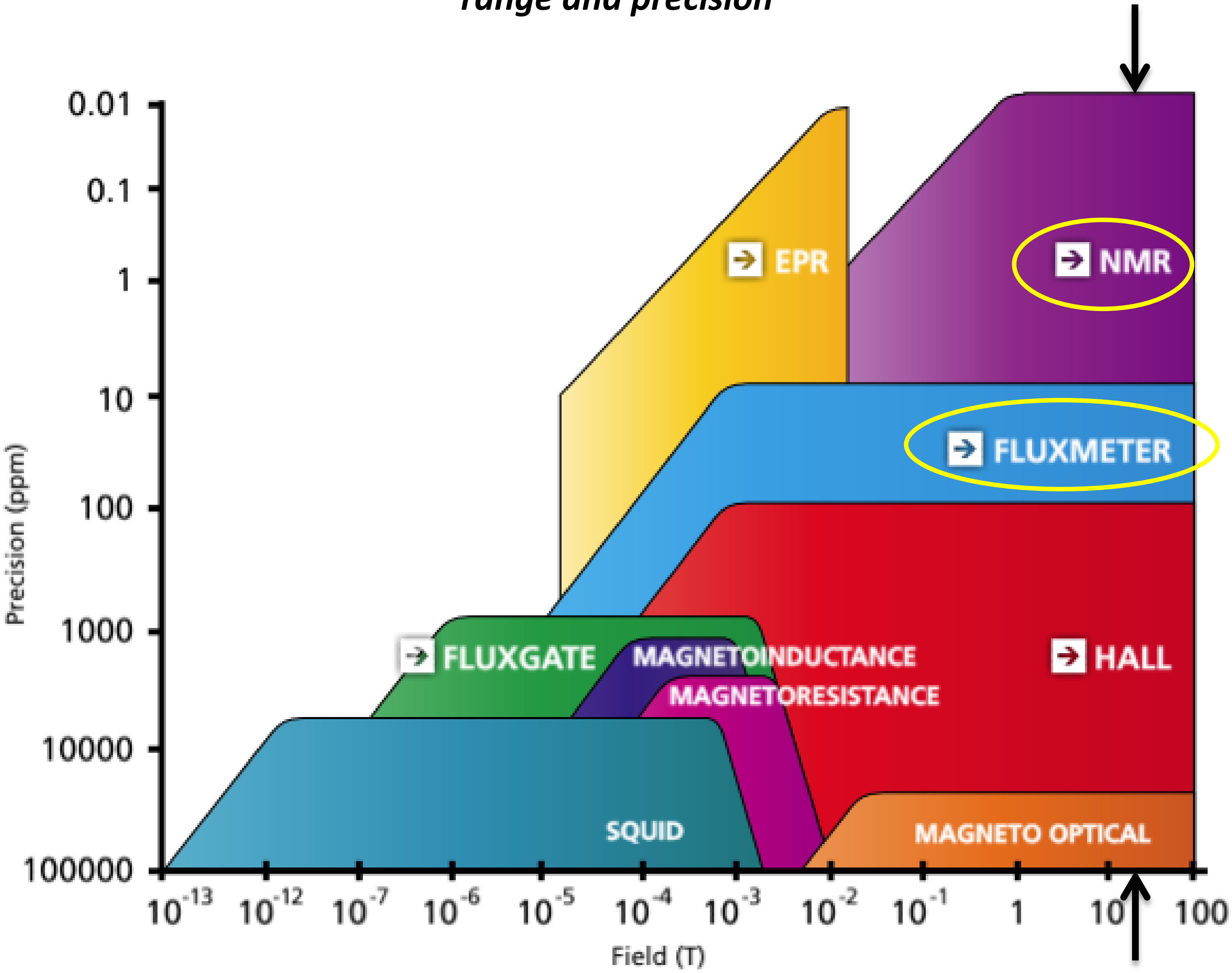
At 37 T: Current density > 30000 A/cm^2

Maximum stress values: 300 MPa

Power density: 2 W/mm^3

Field characterization techniques: overview

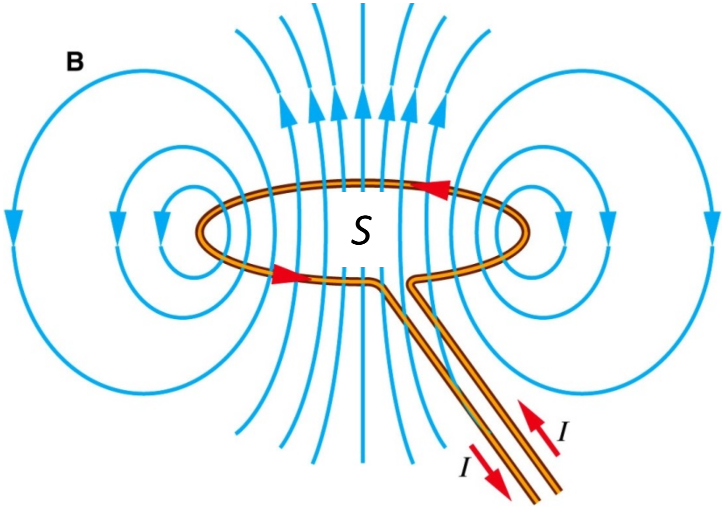
range and precision



© Metrolab S.A.

Fluxmeter measurement principle

Pickup coil



Magnetic induction

$$V = -\frac{d\varphi}{dt}$$

If *B* is homogeneous on the surface

$$V = k\frac{dB}{dt}$$

After integration

$$\Delta B = B_{end} - B_{start} = \frac{1}{k}\int_{t}^{t+\Delta t} V\,dt$$

***k* must be known or calibrated,**
it is the pickup-coil coefficient Vs/T

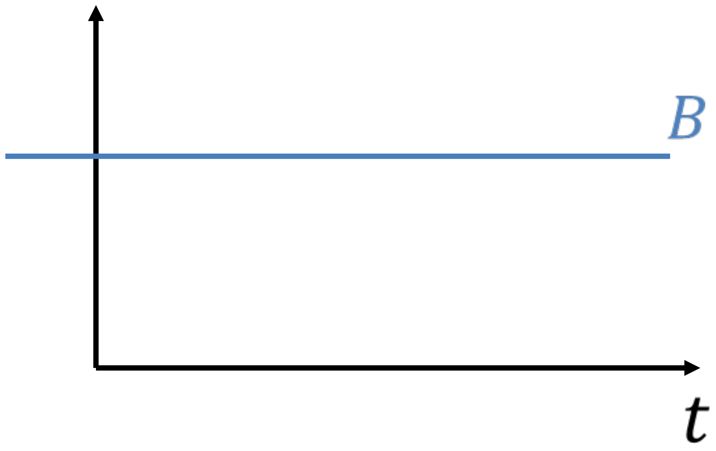
Accuracy of 10 ppm
Spectral bandwidth up to 10^3 kHz

Magnet centering and axial field profile

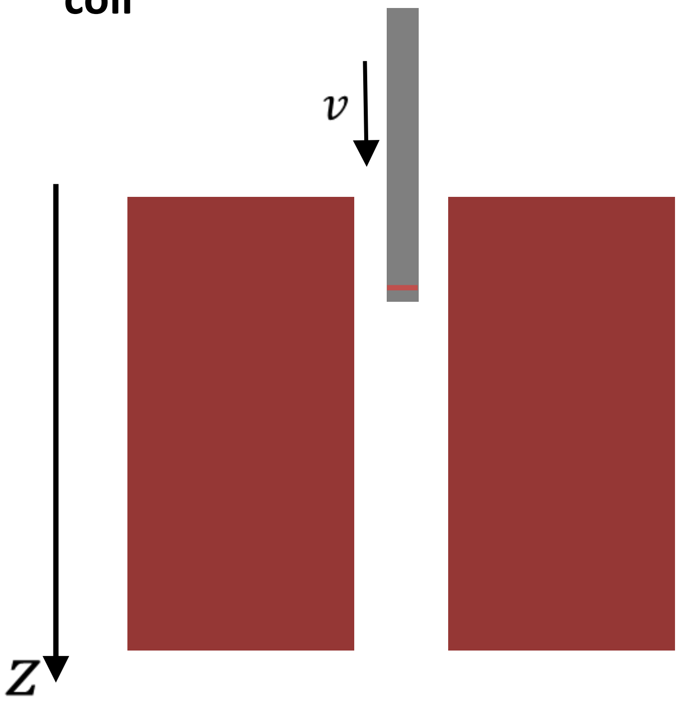
This is a spatial integration over main axis of the magnet.

$$\Delta B = \frac{1}{k}\int_{z}^{z+\Delta z} V\,dz$$

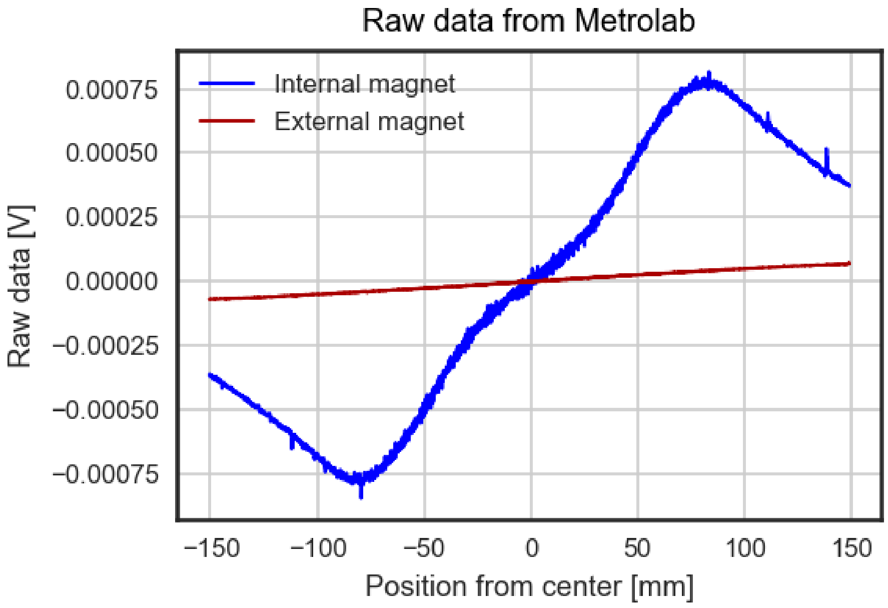
+ constant magnetic field

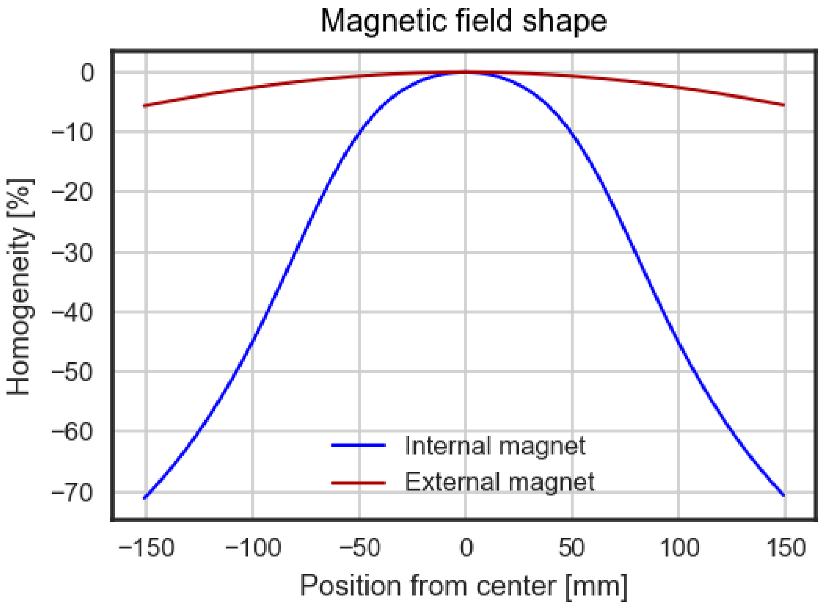


+ Vertical displacement at constant speed of the pick-up coil



Axial field profiles of LNCMI magnets – inner and outer part





Global field profile:

- Available for users on EMFL website.
- Important for modeling of magnet.
- Homogeneity area.
- Levitation zones.

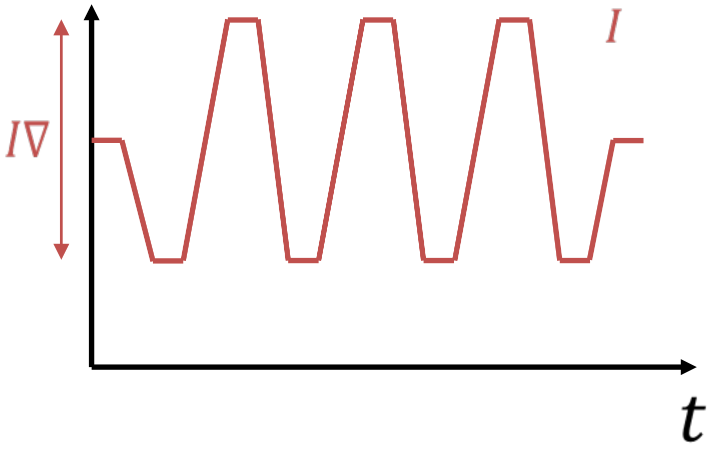
Centering accuracy: 100 μm.

Field factor of the LNCMI magnets

This is a time integration at the center of the magnetic field.

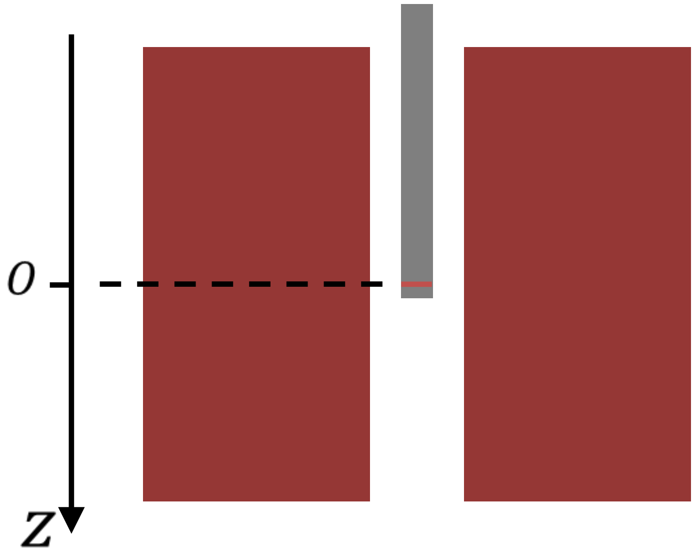
$$\Delta B = \frac{1}{k}\int_{t}^{t+\Delta t} V\,dt$$

+ Multiple sweep in current



ΔI is measured with high precision **DCCT** (Direct Current-Current Transformer)

+ Centered pick-up coil



ΔB is measured

$$FF = \frac{\Delta B}{\Delta I}$$

Field factor accuracy versus NMR: 0.05 %

LNCMI fluxmeter system

Linear piezo motor field mapping system

Spatial resolution:

5 um

Centering accuracy :

100 µm

Magnetic field accuracy versus NMR :

0.05 %

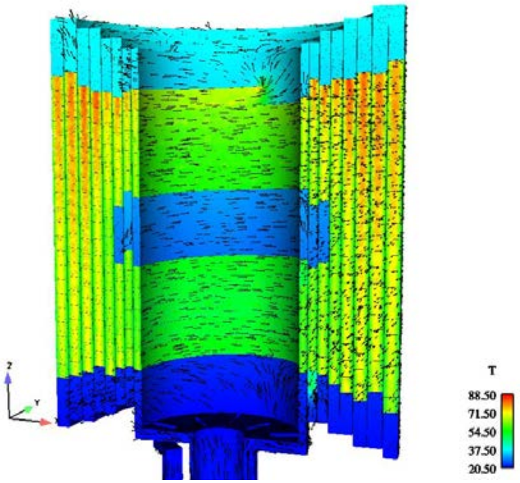
LNCMI efforts for improved field quality – homogenous B

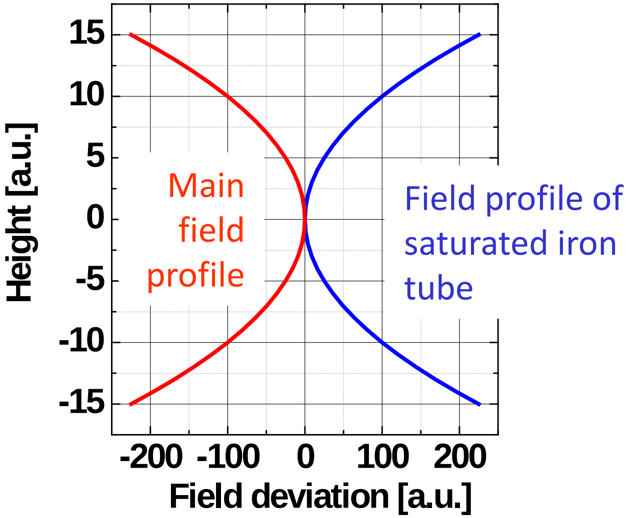
High resolution solid state NMR: Homogeneity of 10^{-6} over the sample (5 mm along axis).

LNCMI two-fold approach

Design of magnets with improved homogeneity

Improvement of homogeneity of existing magnets





- **LNCMI polyhelix magnet technology:**
  Enhanced homogeneity **at all fields**.

✓ Validation by axial (Pick-up coil) and 3d field mapping (NMR)

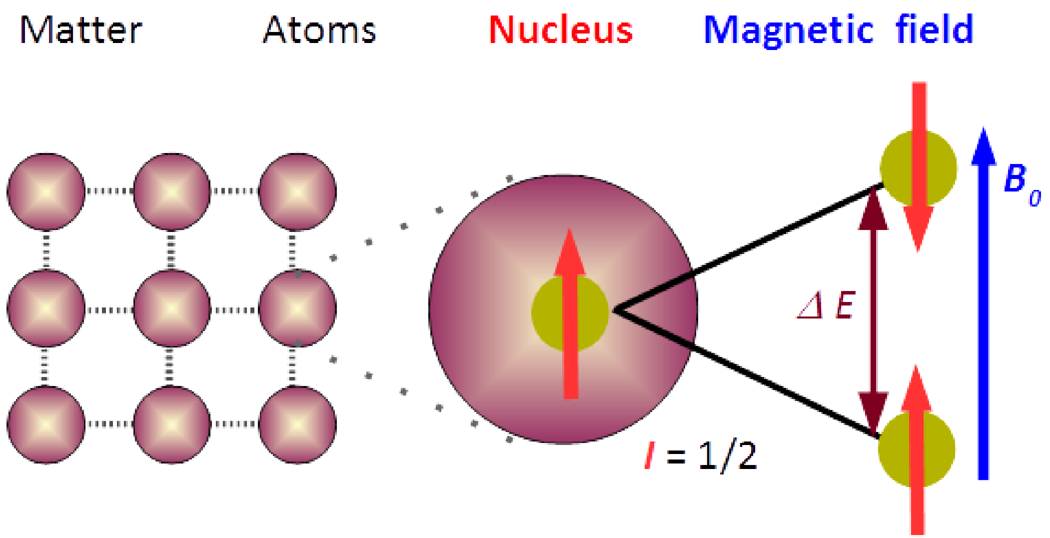
- Active shimming

- **Passive shimming** (iron cylinder):
  Enhanced homogeneity **at one chosen field**.

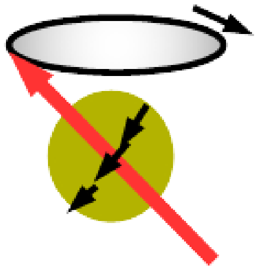
✓ Validation by NMR field mapping:
LNCMI: 10 ppm reached over 5 mm.

Nuclear Magnetic Resonance: precise mesure of B

Quantum effect with classical analogue



Magnetic Torque
$\tau = M \times B_0$



Magnetic energy
$E = -M B_0$

Nuclear angular moment "spin" I
Nuclear magnetic moment M
$M = \gamma I$ <u>*gyromagnetic ratio γ*</u>

Energy difference
$\Delta E = h \gamma B_0$
γB_0 is Larmor frequency

Precession
Larmor frequency
$f = \gamma B_0$

NMR is calibration standard for magnetic field: ppm precision (1 ppm = 10^{-6})

Gyromagnetic ratio γ of ^1H in H_2O at 25°C is CODATA listed: $\gamma = 42.57638507$ MHz/T

However, it requires a "somehow" homogeneous and stable field.

At 35 T Larmor frequency of protons is 1.5 GHz

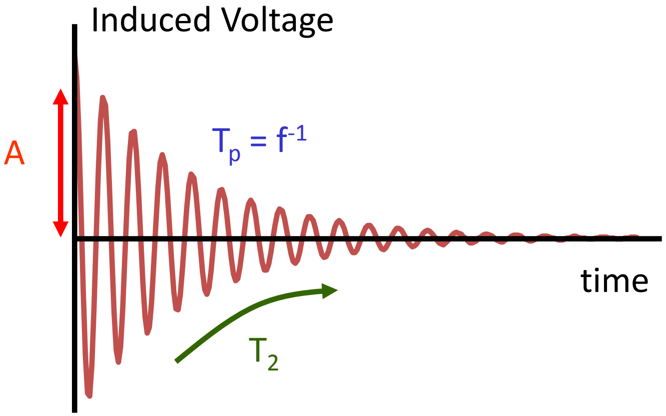
How to measure field by NMR?

Record individual NMR FIDs and determine

instantaneous magnetic field values by FFT (average of 100 us).

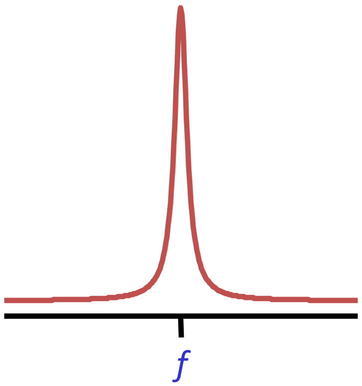
Repeat experiment n times and apply statistical methods for analysis.

Method sensitive to noise related to magnet (vibrations, drift).



Time domain:
“FID”
Duration: 100 μs at 1 GHz / 23.5 T
Limited by field homogeneity.

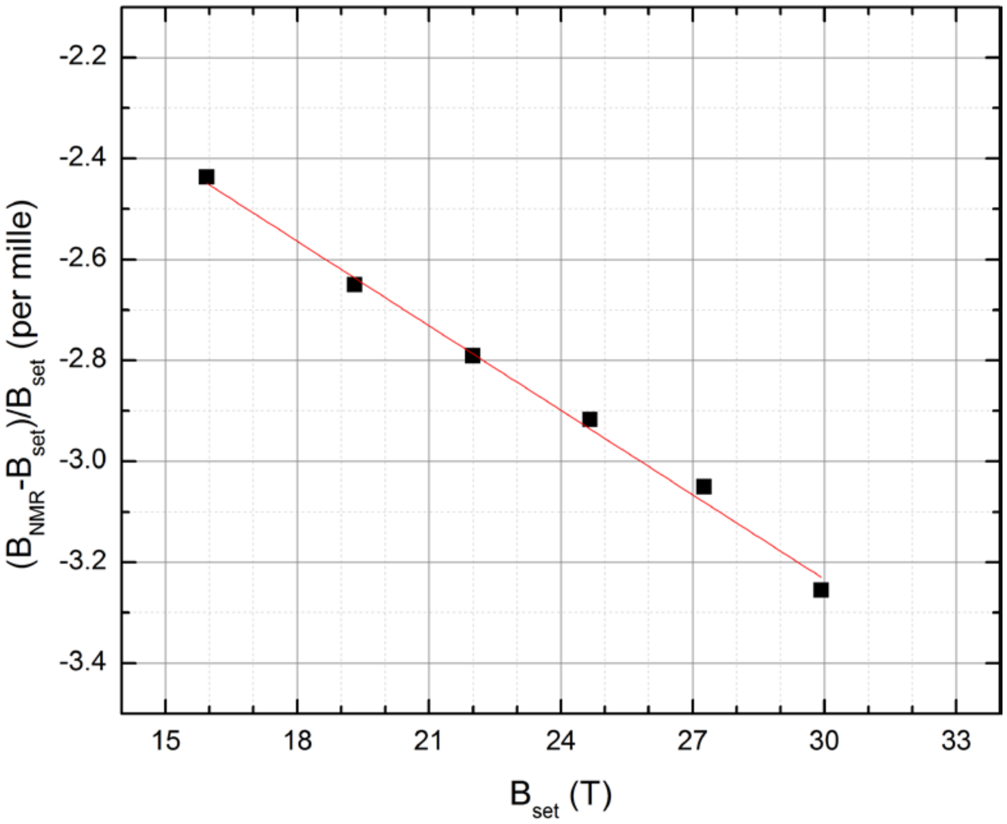
Fourier
transform



Frequency domain:
Spectrum centered at frequency $f = \gamma B$
Width: 10 ppm at 1 GHz / 23.5 T
Accuracy better than width!

Actual project: NMR correction of field factor

Deviation from nominal value



July 2018

M9 Bitter and Helix

^{27}Al NMR at 4.2 K at selected fields

$^{27}\gamma$ = 11.1122 MHz/T

$$B_{NMR} = (1 + a)B_{set} + bB_{set}{}^{2}$$

a = -0.16 %	static error of field factor
b = -5 x 10^{-5} T^{-1}	quadratic non-linear term

Field value at 30 T becomes 0.1 T less than indicated.

Quadratic term is property of magnet:

Thermal expansion, current distribution, hydraulic pressure?

NMR field map

3d field mapping probe available at LNCMI

Variation of sample position by guided motion along threads.

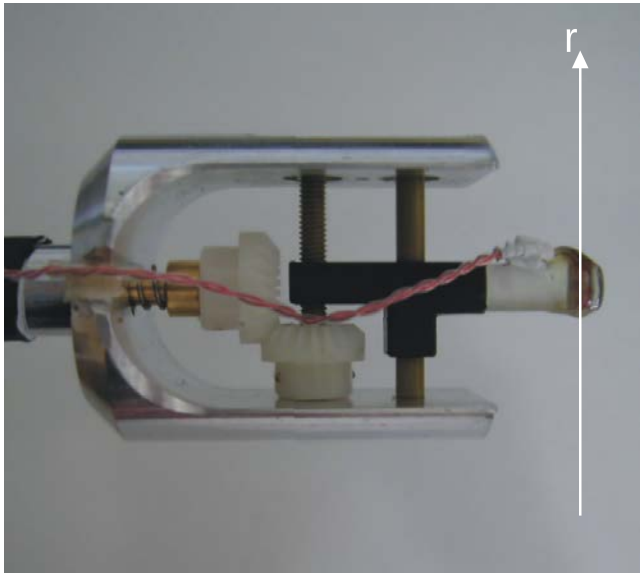
Mapping of field profile in cylidrical coordinates.

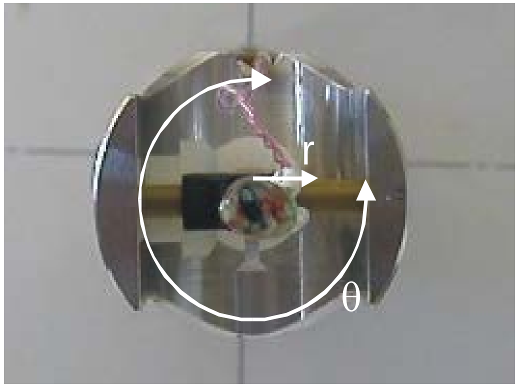
Paths:

$B_z(z, r=0, \theta=0)$

$B_z(z=z_0, r, \theta=\theta_0)$

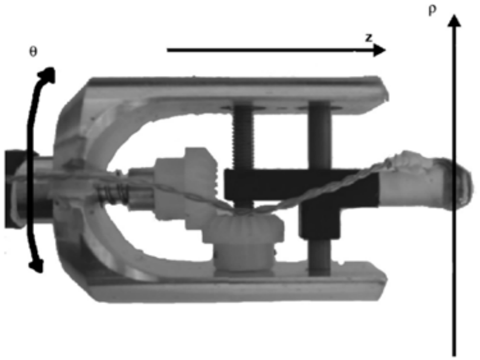
$B_z(z=z_0, r=r_0, \theta)$





3d field mapping for non-axial geometry

Polyhelix magnet with enhanced homogeneity

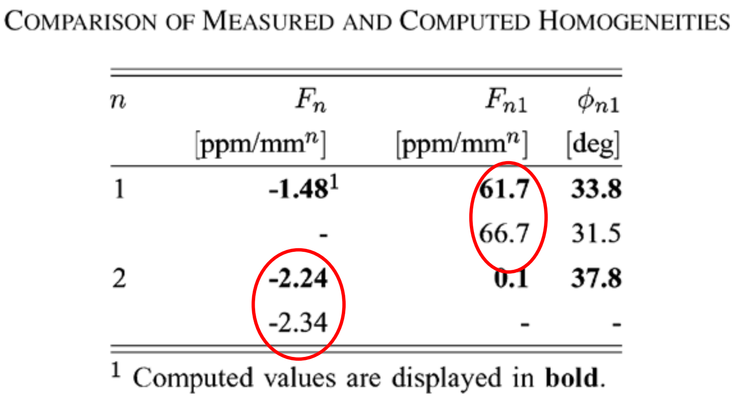


NMR probe
for 3d field mapping

Magnetic field $B_0(\mathbf{r})$ as a function of 3d spatial position was determined by measuring nuclear precession frequency f_0 :$f_0(\mathbf{r}) = \gamma_n B_0(\mathbf{r})$ and the results were compared with numeric calculation:

Validation of 3d numeric analysis

Radial gradient due to non-axial symmetric coils!



COMPARISON OF MEASURED AND COMPUTED HOMOGENEITIES

n	F_n [ppm/mm^n]	F_{n1} [ppm/mm^n]	ϕ_{n1} [deg]
1	**-1.48**[1]	**61.7**	**33.8**
	-	66.7	31.5
2	**-2.24**	**0.1**	**37.8**
	-2.34	-	-

[1] Computed values are displayed in **bold**.

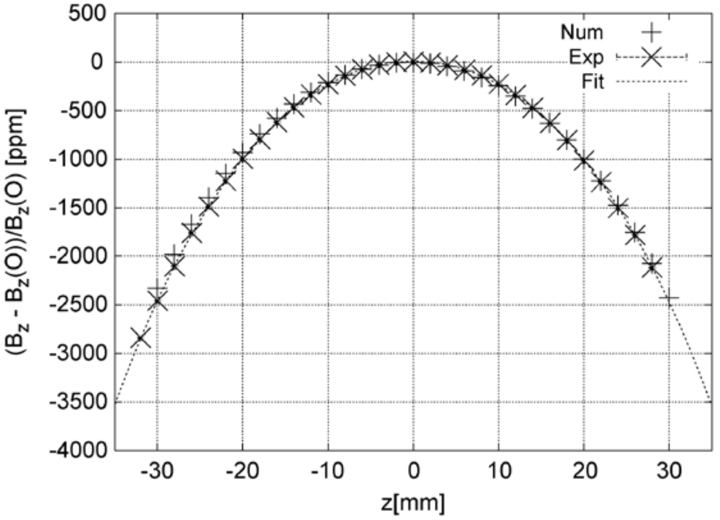


Fig. 2. Magnetic Field Profile along z axis.

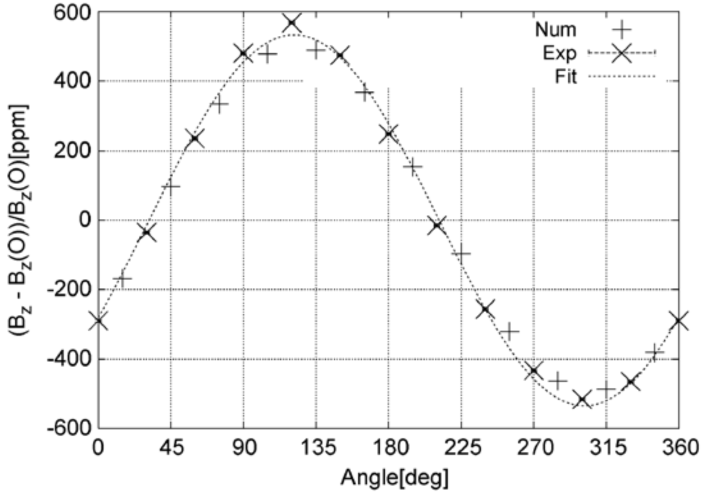


Fig. 3. Azimuthal magnetic field profile at $\rho = 8$ mm and $z = 0$ mm.

C. Trophime et al., IEEE Trans. Appl. Superconductivity, 2006

How to measure time evolution of magnetic field by NMR?

Measure field directly by NMR

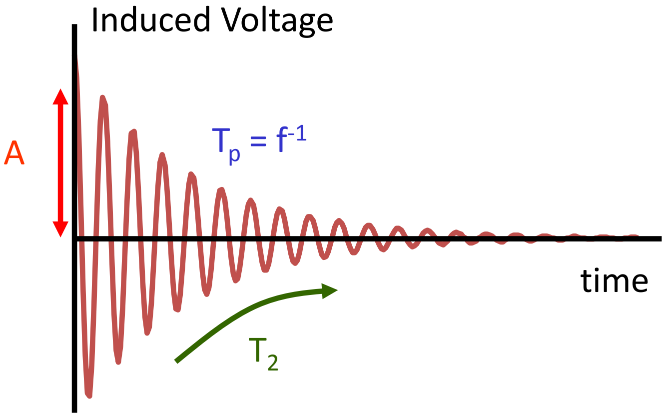
Record individual NMR FIDs and determine

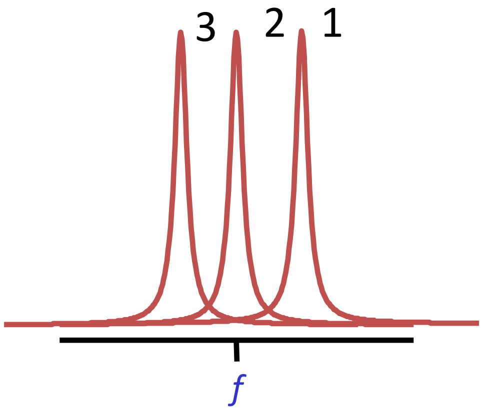
instantaneous magnetic field values by FFT (average of 100 us)

Repeat experiment n times and apply statistical methods for analysis

Bandwidth of method: DC to 10 kHz, ppm precision, lack of spectral resolution

Method sensitive to noise related to magnet (vibrations, drift).





Time domain:
“FID”
Duration: 100 μs at 1 GHz / 23.5 T
Limited by field homogeneity.

Fourier transform →

Frequency domain:
Spectrum centered at frequency $f = \gamma B$
Width: 10 ppm at 1 GHz / 23.5 T
Accuracy better than width!

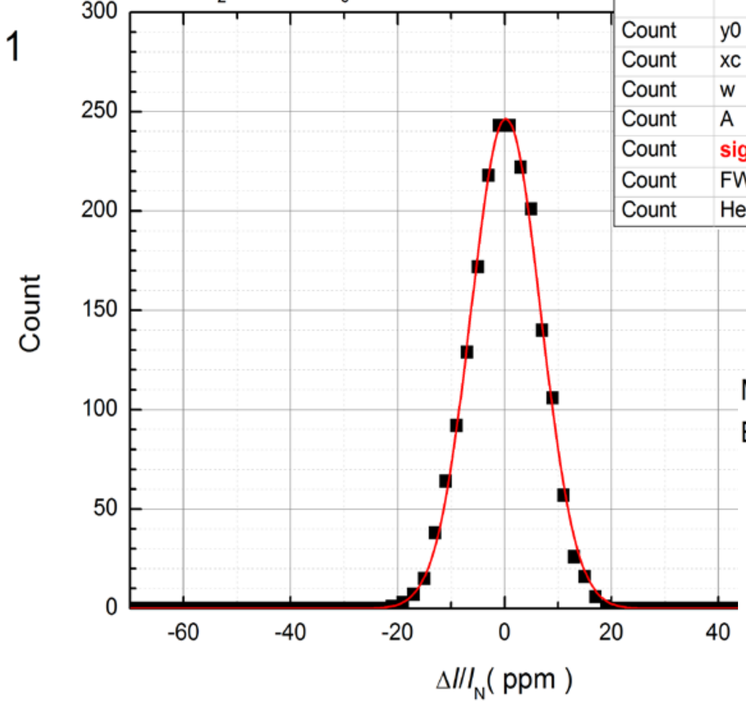
Field properties of installation in June 2017 as seen by NMR

Specification of power supply: 20 ppm of nominal <u>current</u> in band from 0.1 Hz to 10 kHz.

M10 50mm, Group1: 2 x 11800 A on Bitter Group 2, 2 x 10600 A on Helix

B = 23.5324 T, ^{1}H in H_2O.GdCl, f_0 = 999.885 MHz

1



		Value	Standard
Count	y0	-0.12224	0.26618
Count	xc	0.23999	0.03764
Count	w	13.02403	0.07785
Count	A	4024.447	22.15127
Count	sigma	6.51202	
Count	FWHM	15.33463	
Count	Height	246.5476	

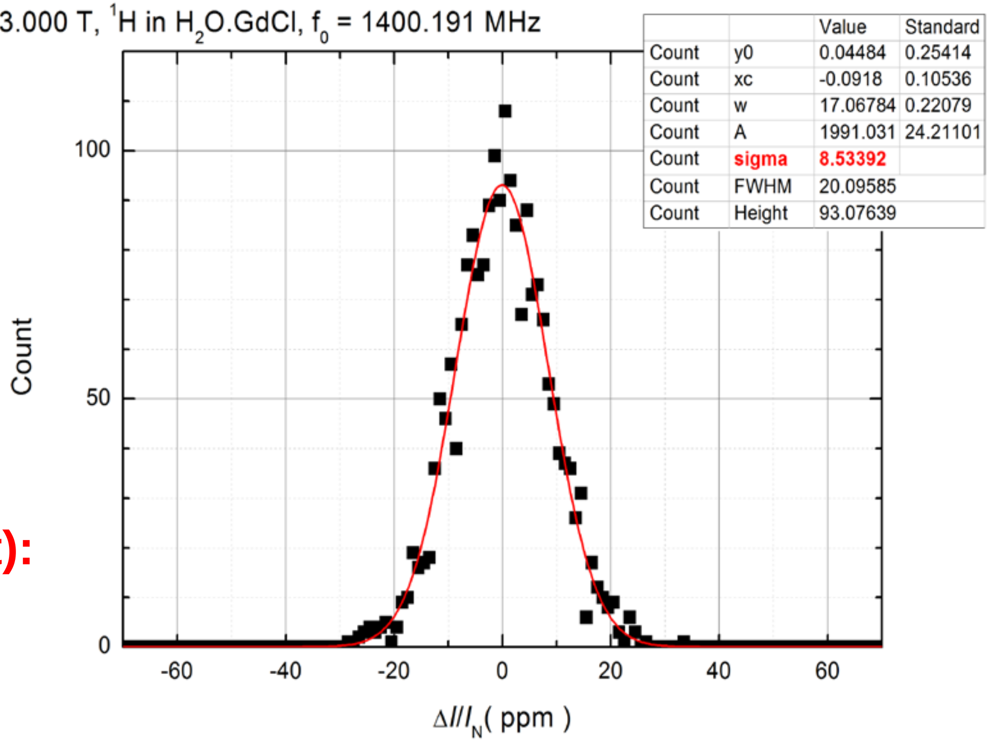
M10 at 24 T (current + magnet):

3 σ = 19.5 ppm

M9 34 mm, Group1: 2 x 13900 A on Helix Group 2, 2 x 13900 A on Bitter

B = 33.000 T, ^{1}H in H_2O.GdCl, f_0 = 1400.191 MHz

3



		Value	Standard
Count	y0	0.04484	0.25414
Count	xc	-0.0918	0.10536
Count	w	17.06784	0.22079
Count	A	1991.031	24.21101
Count	sigma	8.53392	
Count	FWHM	20.09585	
Count	Height	93.07639	

M9 at 33 T (current + magnet):

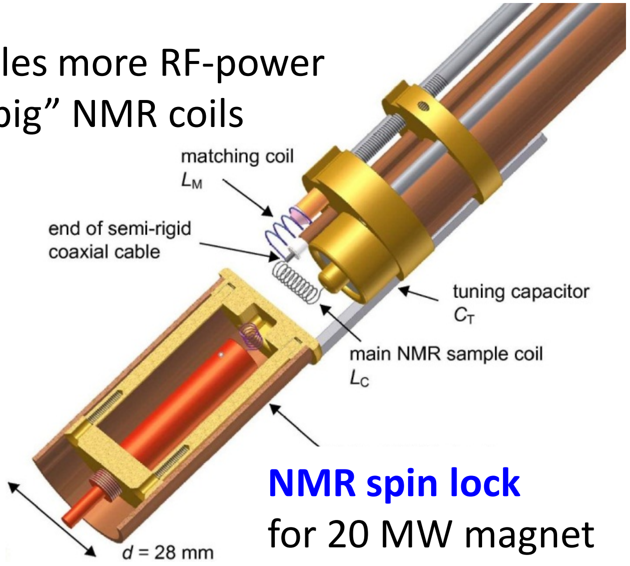
3 σ = 25.5 ppm

Enhanced field stability and resolution for 20 MW magnets

NMR spin lock and Ferroshim: 10 ppm / Ø = 5 mm

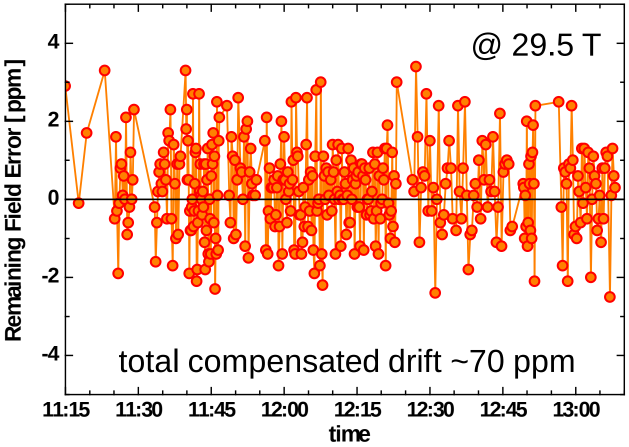
New NMR RT probe for 20 MW magnet

handles more RF-power
for “big” NMR coils



NMR spin lock
for 20 MW magnet

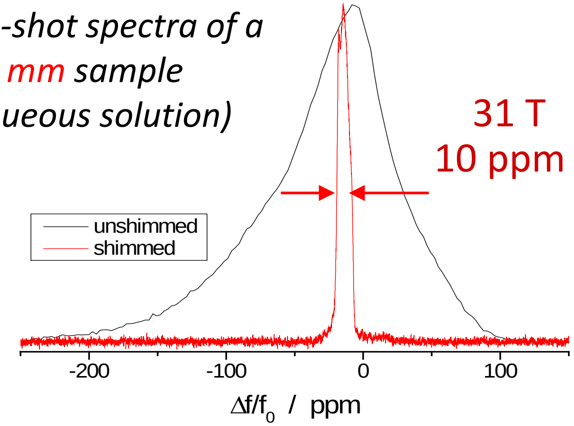
to cancel the **field drift**:

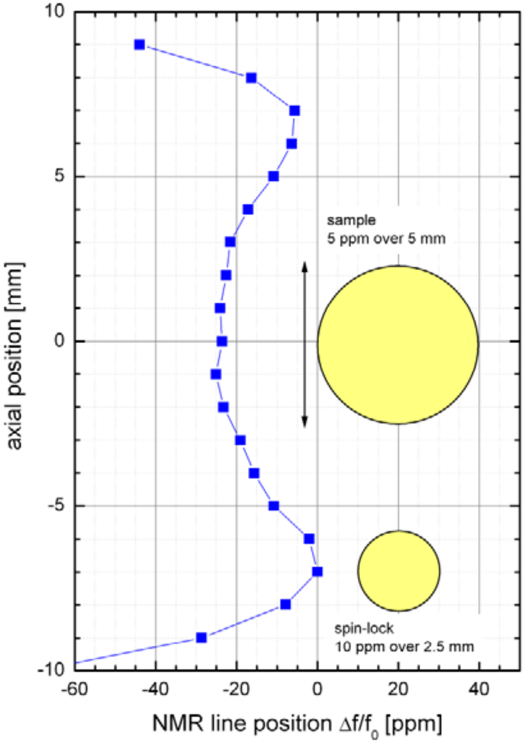


Further steps:
Active compensation
of the field fluctuations
Homogeneous, > 30 T magnets

Passive ferroshim:

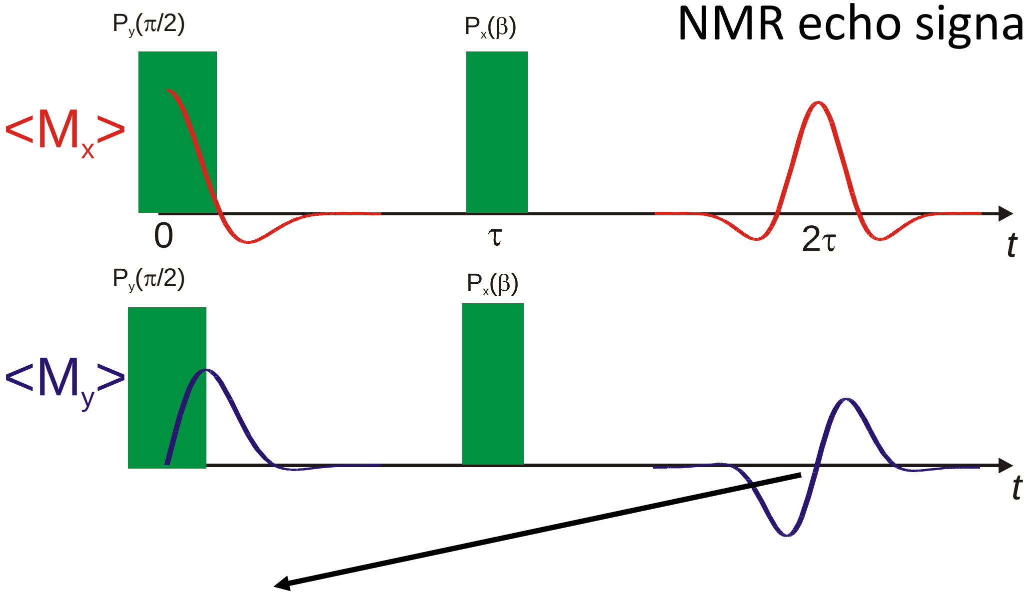
^{23}Na single-shot spectra of a standard 5 mm sample (NaCl in aqueous solution)





NMR stabilization of resistive magnets – spin echo method

Spin-lock has to work in inhomogeneous field



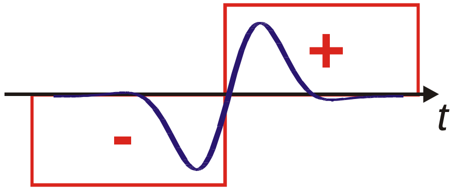
NMR echo signal

Frequency offset: $\Delta\omega=(\omega-\omega_0)$

Frequency spectrum: $I(\omega)$

$$\int I(\Delta\omega)\cos(\Delta\omega(t-2\tau))\,\mathrm{d}\omega$$

$$\int I(\Delta\omega)\sin(\Delta\omega(t-2\tau))\,\mathrm{d}\omega$$



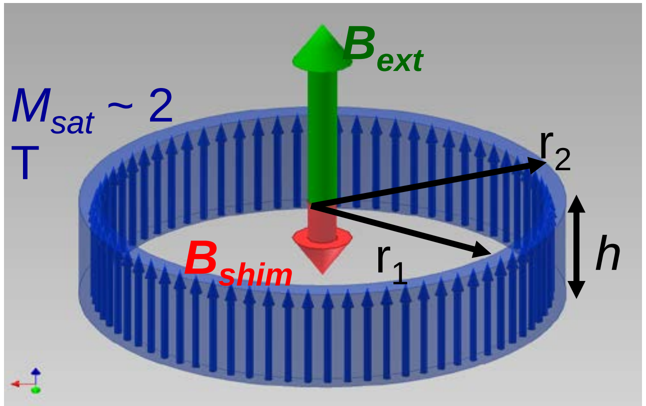
$$G(\omega_0)=\int_{2\tau\pm t_1}\left(\int_{\Delta\omega}\mathrm{sign}(t-2\tau)I(\Delta\omega)\sin(\Delta\omega(t-2\tau))\,\mathrm{d}\omega\right)\mathrm{d}t$$

$G(\omega_0)$ changes sign if ω_0 is first moment of $I(\omega)$: control signal

Signal enhancement by CPMG

Passive shimming of resistive magnets

permanent magnets in electromagnets



Ferromagnetic cylinder (iron):

Saturated ferromagnet (M_{sat} ~ 2 T) generates its own intrinsic magnetic field B_{shim} .

Center region: B_{shim} in opposition to external field B_{ext}:

$$B_{z,\mathrm{shim}}(z,r=0) = -\frac{M_{sat}}{2}\left[-\frac{z-l}{\sqrt{r_1^2+(z-l)^2}}+\frac{z-l}{\sqrt{r_2^2+(z-l)^2}}\right]_{l=-h/2}^{l=+h/2}$$

Near center:

Inhomogeneity of the external field B_{ext} will be reduced.

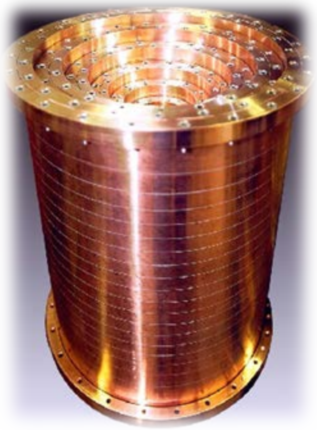
Optimized homogeneity

obtained at only one particular field value B_{ext}

and with a appropriate design of the shim itself (height and thickness).

and with a precise (sub-mm) positioning of shim in field.

Magnetometry at LNCMI



- **Science in high magnetic fields:**
  - *Metrology – precise value of B*

    NMR: precise knowledge of B is mandatory for precise NMR
  - *Spatial field distribution – precise distribution of B*

    NMR: resolution

    Magnetization: magnetic torque, magnetic force

    Levitation: magnetic force
  - *Time variation of magnetic field – drift and spectrum of B(t)*

    NMR: resolution

    All other techniques: noise

- **Development of high field magnets and power installation:**
  - *Safety and centering of magnets*

    Forces

    Optimal field profiles
  - *Validation of field calculations:*

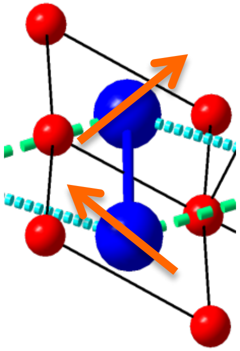
    Homogeneity

    Levitation zones
  - *Improvement of field quality*

    Spatial resolution

    Temporal stability

Matter and magnetic field

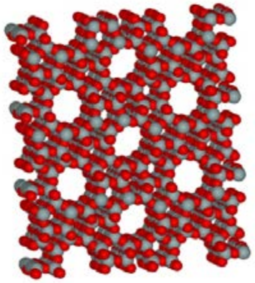


Field induced phenomena (quantum effects)

Phase transitions and new states of matter:

2d electrons, finite electron systems (nanostructures), superconductivity, heavy fermions, low dimensional conductors, low dimensional magnets.

Fundamental: **condensed matter physics**

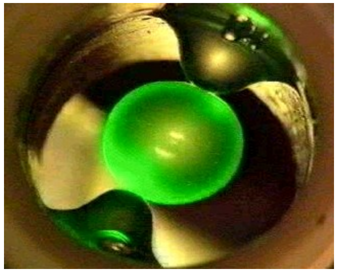


"Smooth B properties of matter" (classical and quantum)

Spectroscopy under variable B:

NMR, EPR, optics, neutrons, x-ray, thermodynamics and transport under B: magnetization, calorimetry, electric and heat transport

Fundamental and applied : **physics, chemistry, biology, medicine**



Classical electrodynamics (magnetic force)

Magnet design, magnetohydrodynamics, magnetic alignment, magnetic confinement, magnetic levitation

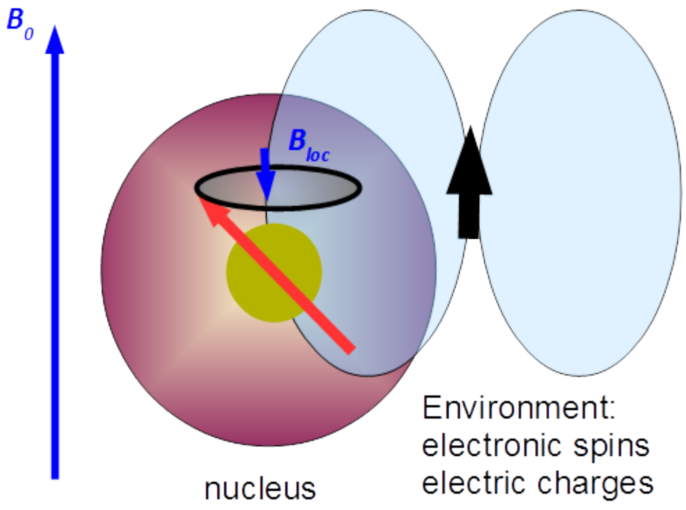
Fundamental and applied: **soft matter, biology, plasma, energy, metallurgy**

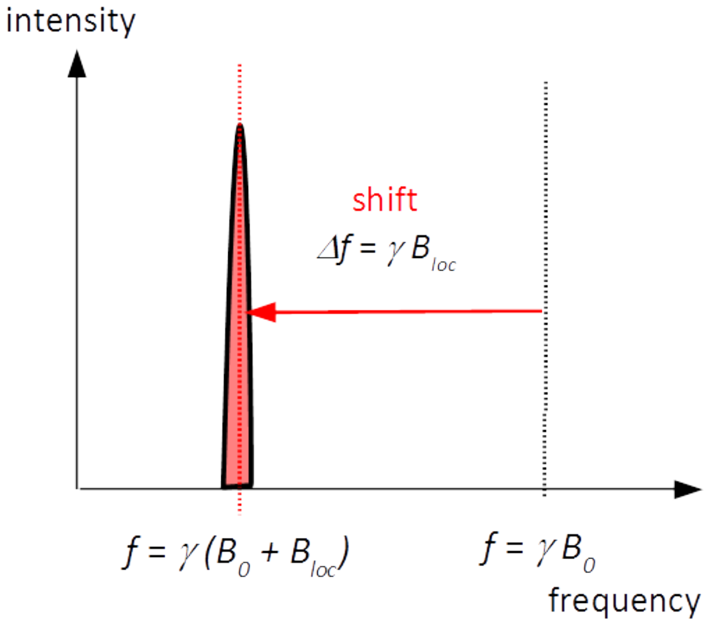
Science in high magnetic fields at LNCMI Grenoble

		Maximum Field	**Temperature range**
Magnetotransport	DC, AC, pressure, contactless	37 T	20 mK – 300 K
Magnetization – dHvA	**Cantilever** **Faraday balance**	37 T	20 mK – 300 K 1.2 K – 300 K
Specific heat		30 T	1.2 K – 300 K
“Thermotransport”		35 T	300 mK – 300 K
Magnetic Resonance	**NMR in solid state physics** **High resolution NMR (chemistry)** EPR (superconducting magnets)	35 T 30 T (fix) 16 T	50 mK – 300 K 300 K 1.5 K – 300 K
Ultrasound attenuation		35 T	1.5 K – 300 K
Magneto-optics	Photoluminescence, μ-PL Raman, μ-Raman Far Infrared Spectroscopy	30 T 30 T 35 T	1.5 K - 300 K 1.5 K – 300 K 1.5K - 300 K
Applied Superconductivity	**Critical current** **Coil test in 170 mm bore size magnet**	30 T 18 T	4.2 K - T_c 4.2 K
Magneto-science	Chemistry, **Orientation of ferromagnets, Levitation**	10 T (380 mm) 35 T (34 mm)	

NMR – what for?

Nuclear spins as monitors in matter





Many identical nuclear spins act as local “observers”.

Concept of additional local magnetic field seen by nucleus B_{loc}

Standard NMR:

B_{loc} proportional to B_0 , aligned and very weak, ratio of 10^{-6} (ppm) to 10^{-9} ppb

Precise magnetic field: superconducting magnets

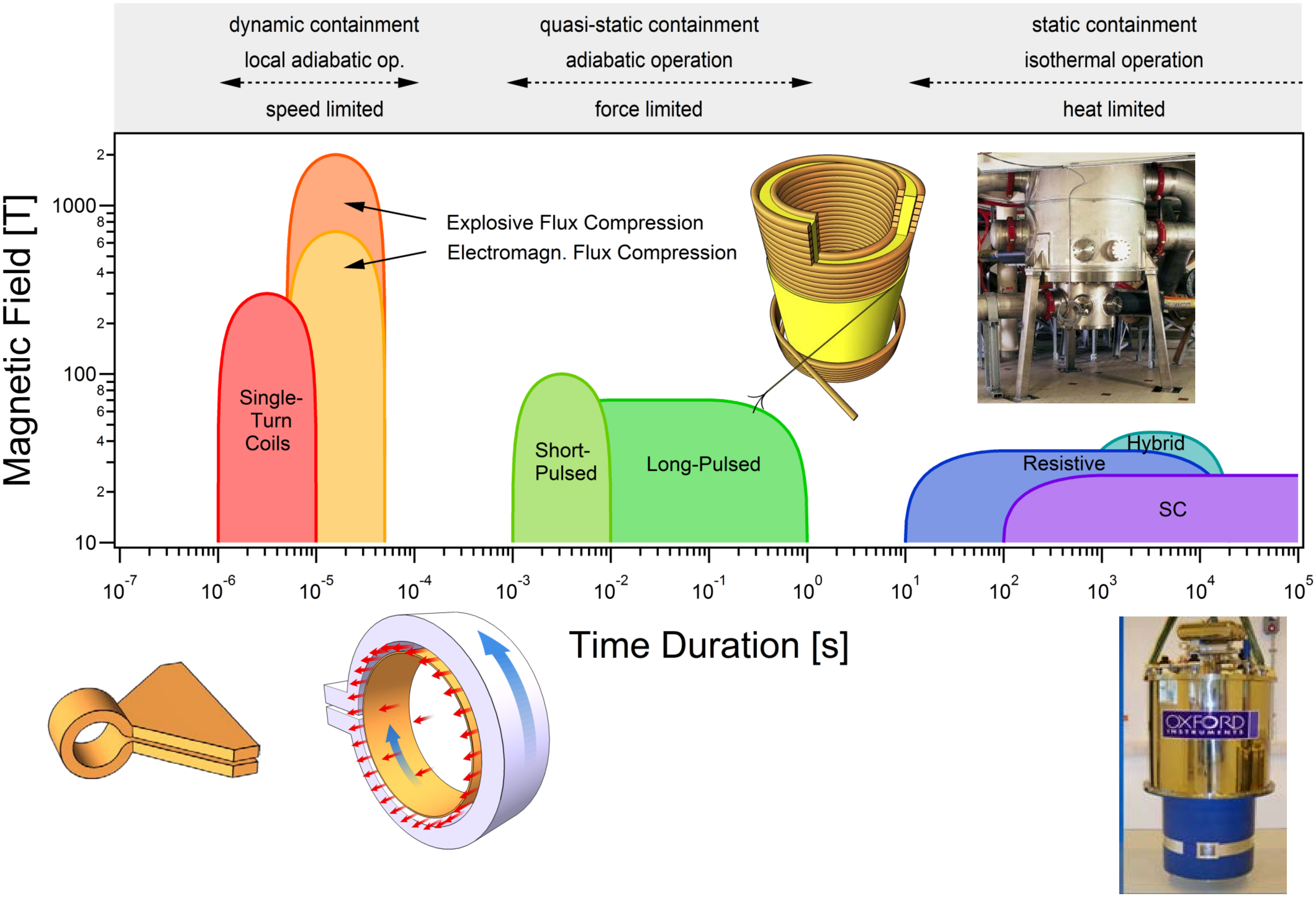
Extreme NMR:

Additional terms in B_{loc}, sometimes not proportional and aligned.

From weak to strong, i.e. $B_{loc} > B_0$

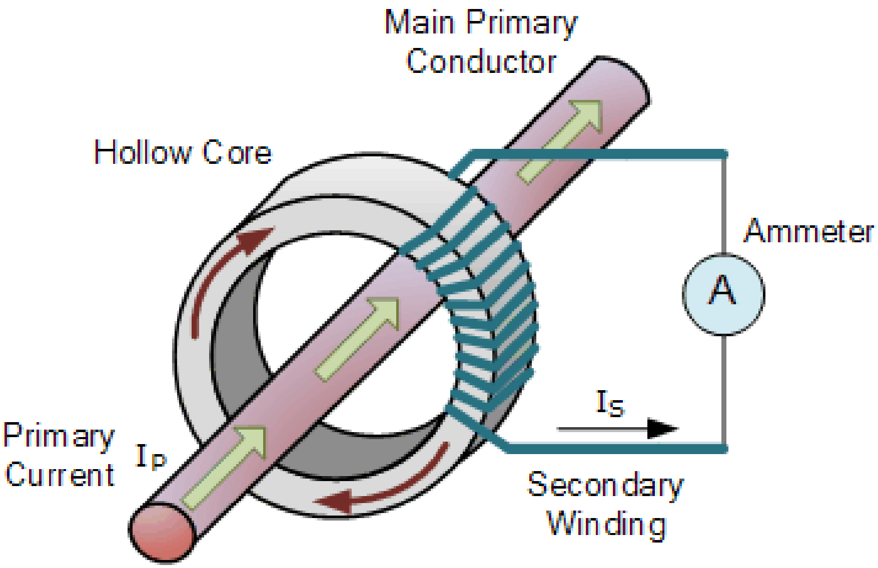
Precise knowledge of external field B_0 is mandatory for precise NMR studies

Types of magnetic fields

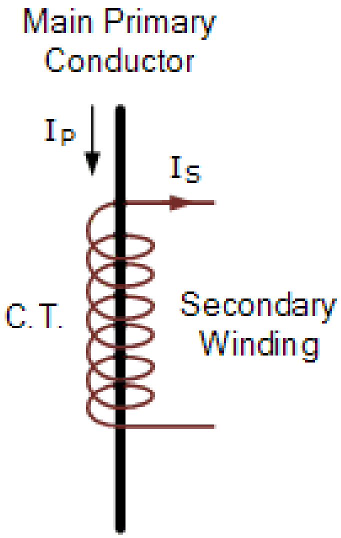


Current Measurement and Regulation: DCCT

developed and optimized by CERN



Construction



Circuit Symbol

<u>DCCT = Direct Current Current Transformer</u>

- Primary current generates magnetic field in hollow core: one conductor, up to 16500 A
- Compensation by secondary current: many turns, 1 A typically
- Detection of “zero flux” by Hall probe or ac-method
- Calibration provides precise and fast absolute current measurement:
  10^{-6} = ppm (parts per million) range, kHz bandwidth

Magnetic field production - electromagnets

Biot Savart law:

$$\mathbf{B}(\mathbf{x}) = \frac{\mu_0}{4\pi}\int \mathbf{J}(\mathbf{x}')\times\frac{\mathbf{x}-\mathbf{x}'}{\left|\mathbf{x}-\mathbf{x}'\right|^3}d^3x'$$

Magnetic force (Lorentz force):

$$\mathbf{F} = \int \mathbf{J}(x)\times\mathbf{B}(\mathbf{x})d^3x$$

Magnetic energy:

$$E = \frac{1}{2\mu_0}\int \mathbf{B}^2(\mathbf{x})d^3x = \frac{1}{2}L\,I^2$$

coil with inductance *L* and current *I*

Magnetic pressure (energy density):

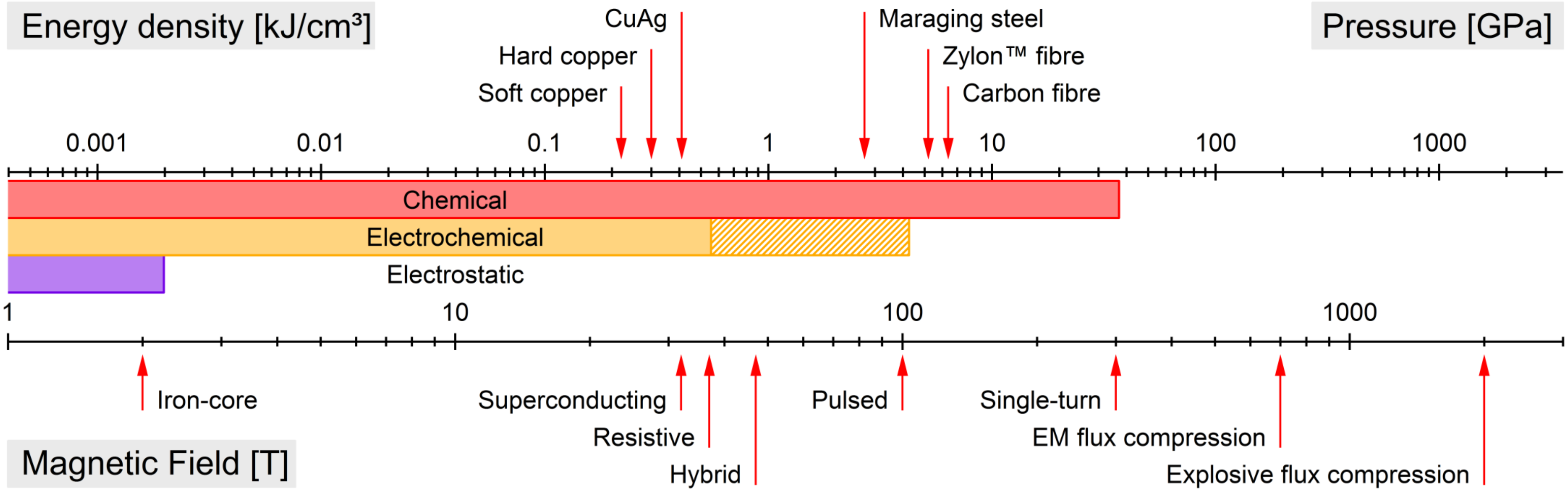
$$p = \frac{\mathbf{B}^2(\mathbf{x})}{2\mu_0}$$

Power dissipation:

$$P = \int \rho(x)\,\mathbf{J}^2(x)d^3x \propto \mathbf{B}^2$$

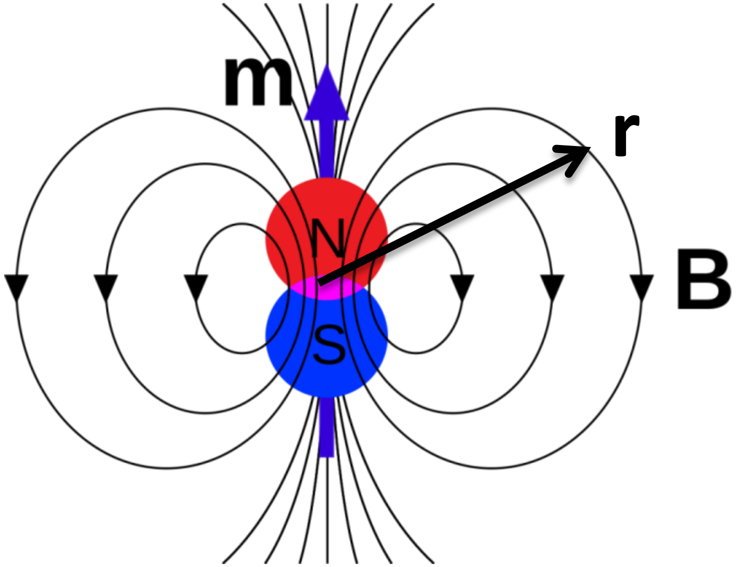
ρ specific resistance

Magnetic field production - electromagnets



Origin of magnetic field – charge and spin

$$\mathbf{B}(\mathbf{r}) = \frac{\mu_0}{4\pi}\left[\frac{3\hat{\mathbf{r}}\left(\hat{\mathbf{r}}\cdot\mathbf{m}\right)-\mathbf{m}}{|\mathbf{r}|^3}\right]$$



Dipole field: important in atomic and molecular physics.

Magnetic dipole in **m** external magnetic field B

Magnetic torque

$$\mathbf{N} = \mathbf{m}\times\mathbf{B}$$

Magnetic energy of dipole in external field

$$E = -\mathbf{m}\cdot\mathbf{B}$$

Magnetic force

$$\mathbf{F} = \nabla(\mathbf{m}\cdot\mathbf{B})$$

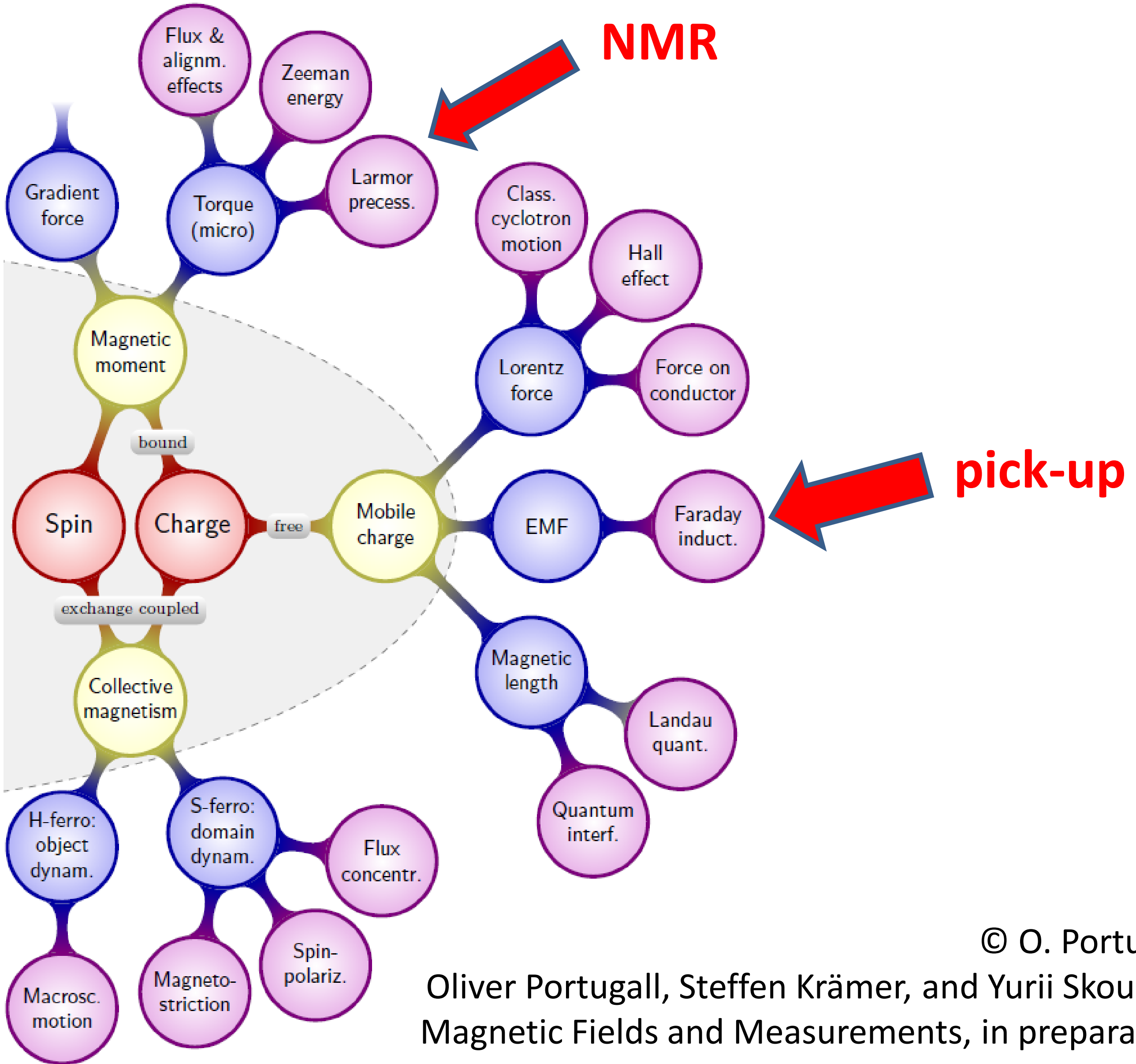
only present in inhomogeneous magnetic fields

Field characterization: Overview and methods

physical observable
measurement technique

physical quantity

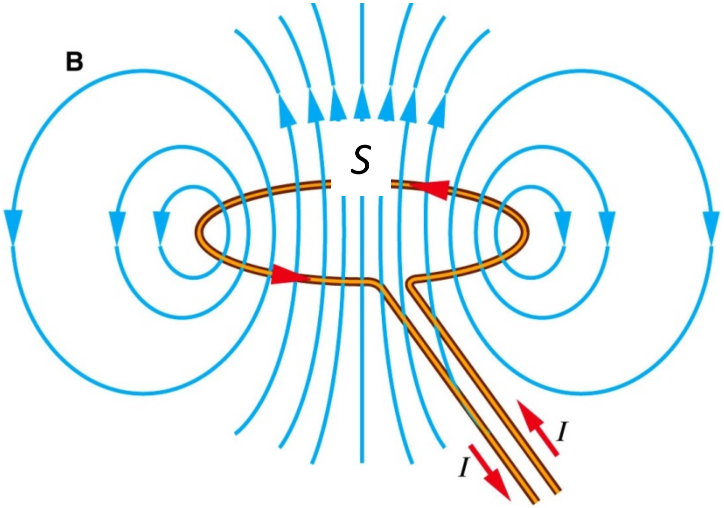
atomic property



© O. Portugall
Oliver Portugall, Steffen Krämer, and Yurii Skourski,
Magnetic Fields and Measurements, in preparation

Field characterization techniques: Fluxmeter by pick-up

Induction coil placed in magnetic field



magnetic flux $$\varphi = \int \boldsymbol{B} \cdot d\boldsymbol{S}$$

induction law $$V = -\frac{d\varphi}{dt}$$

$$\varphi_{end} - \varphi_{start} = \int_{t_{start}}^{t_{end}} V \cdot dt$$

needs precise integrator

$$B_{end} - B_{start} = \frac{\varphi_{end} - \varphi_{start}}{k}$$

needs profile dependent calibration

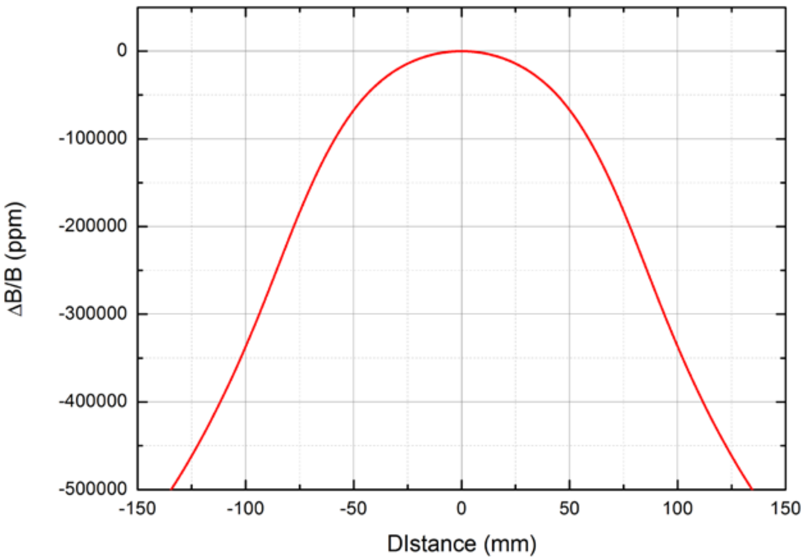
$k = S$ for homogeneous field

Accuracy of 10 ppm

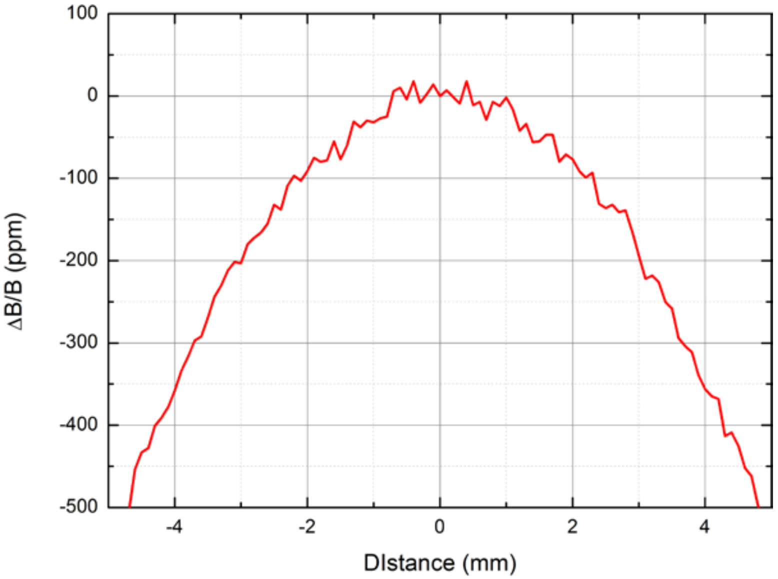
Spectral bandwidth up to 10^3 kHz

Continuous sampling provides power spectrum

Field profiles of LNCMI M9 magnet

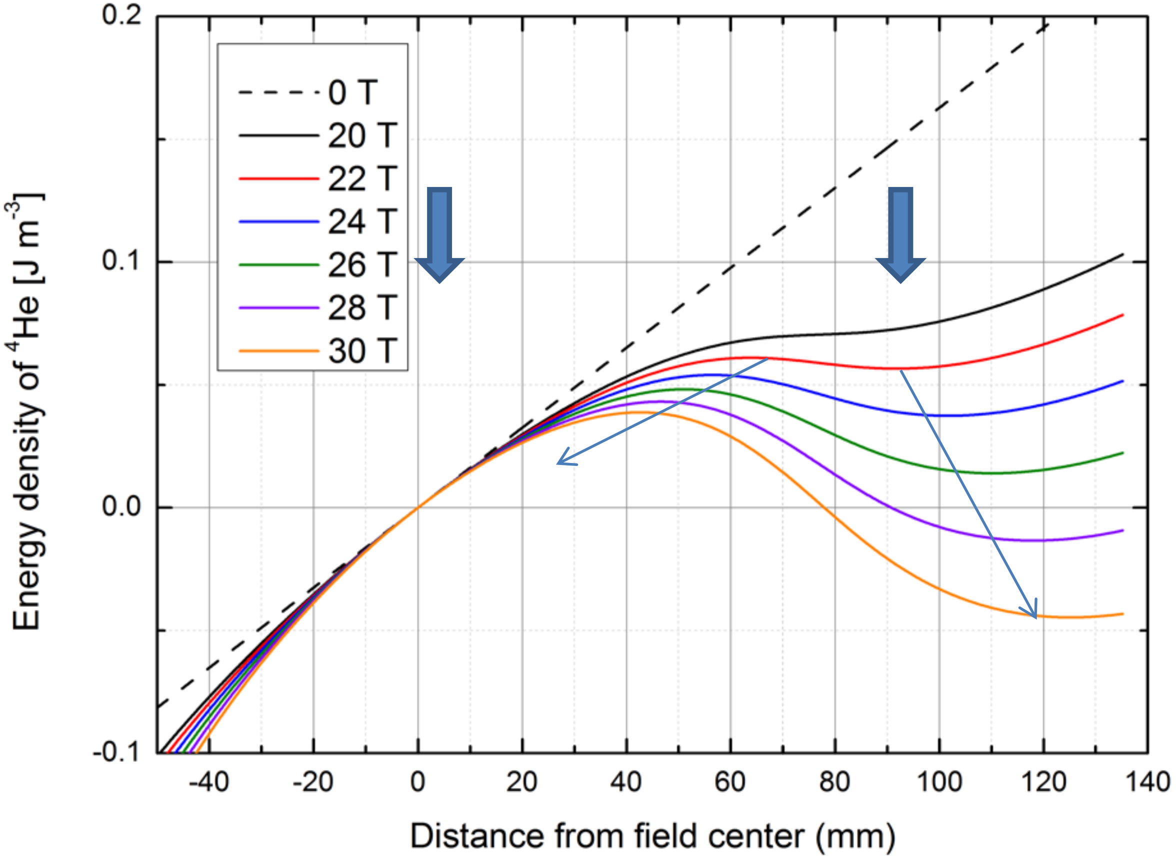


Global profile:
Important for modeling of magnet.
Definition of levitation areas.
Available for users on EMFL website.



Field profile near center:
Important for positioning of user experiments.
Field homogeneity In 1 cm DSV
Here: ΔB/B = 500 ppm

Application of axial field map: diamagnetic levitation of helium



Energy of ^{4}He in presence of magnetic field.

Gravitational energy:

$$E_{grav} = \rho g z$$

Magnetic Energy:

$$E_{mag} = -\frac{1}{2\mu_0}\chi_m B(z)^2$$

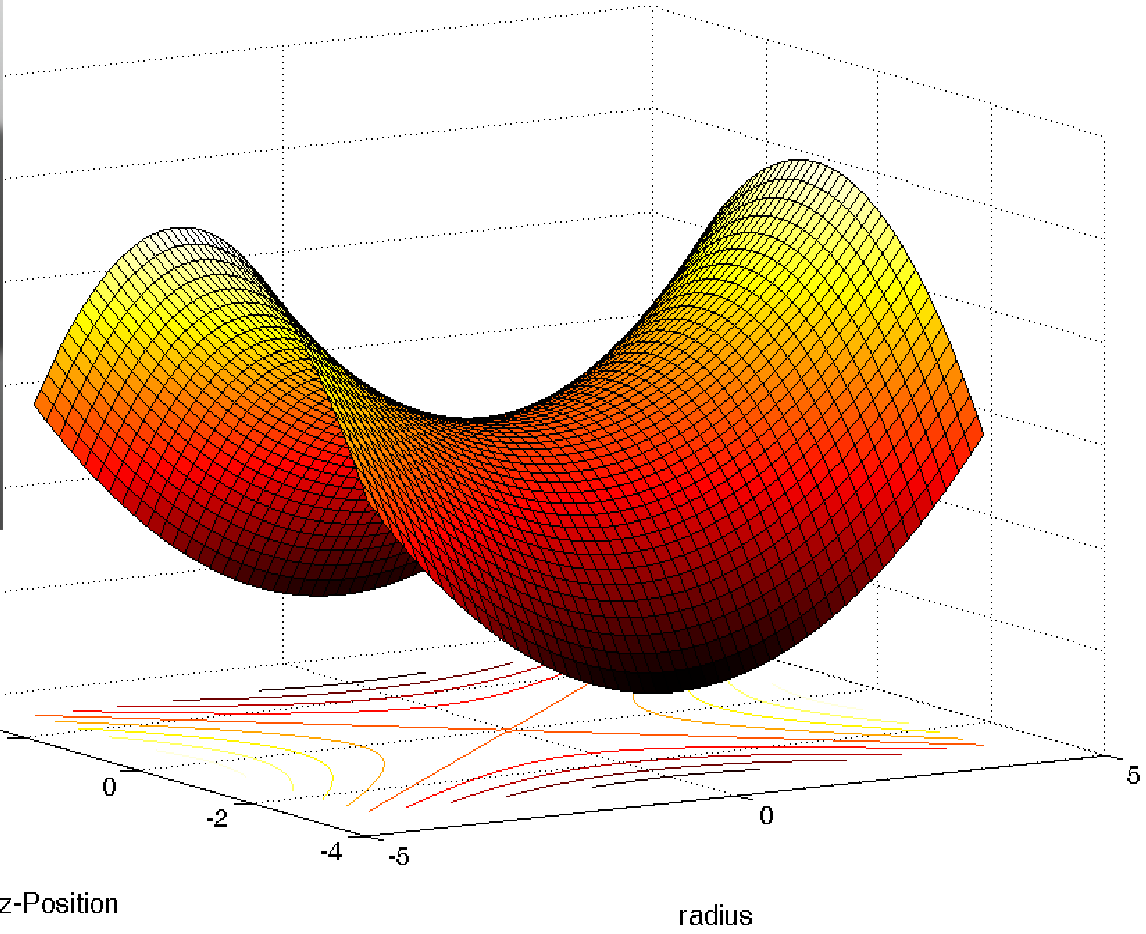
$$E = E_{grav} + E_{mag} = \rho g z - \frac{1}{2\mu_0}\chi_m B^2 = \rho g \left(z - \frac{\chi_m}{2\mu_0 g \rho} B(z)^2 \right)$$

$$\frac{\chi_m}{\mu_0 g \rho} = 4.824 \text{ x } 10^{-4} \text{ m T}^{-2}$$

Levitation zone above threshold field near 22 T for LNCMI M9 magnet.

S.K.,K.P. and Julien Jousset, 2018

3d spatial field distribution



Simplest model:

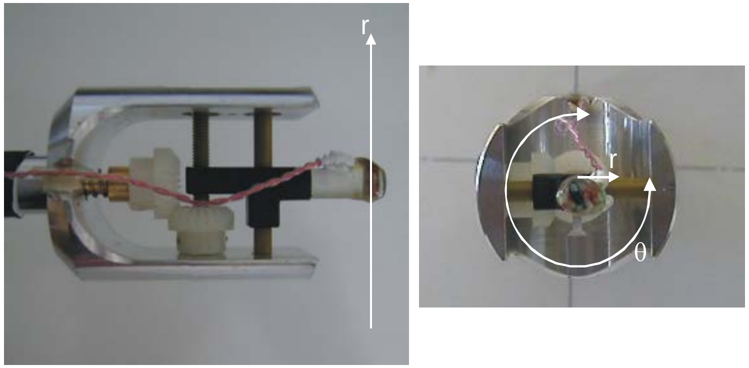
LNCMI M9 14 helix axial symmetry. Principal inhomogeneity: **z2 term**

$$B(z,r) = B_0 + G_{z2}\,(z^2 - 0.5\,r^2)$$

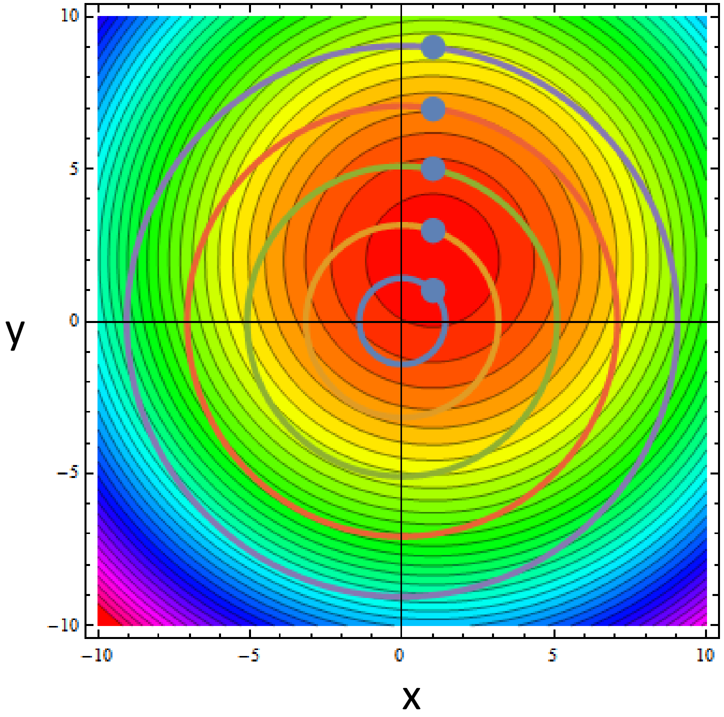
NMR and pick-up: **G_{z2} up to -25 ppm/mm^2**

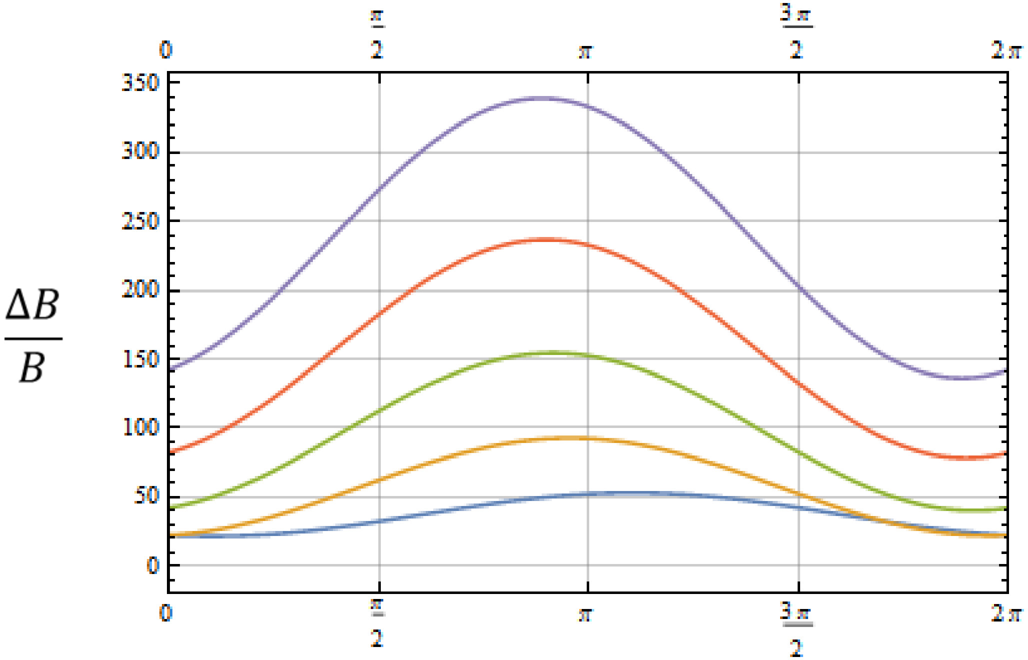
Radial centering of magnets

Off-center effects in axial symmetry



Mapping path along
concentric circles with increasing radius.
Fitting of data using simple model provides center.

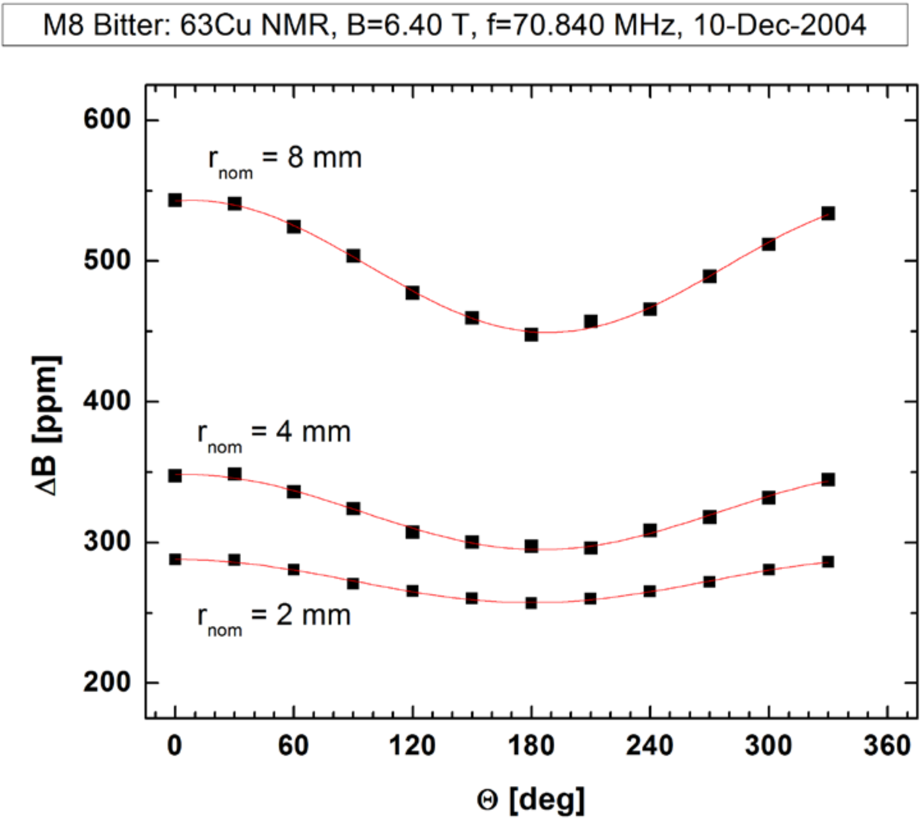


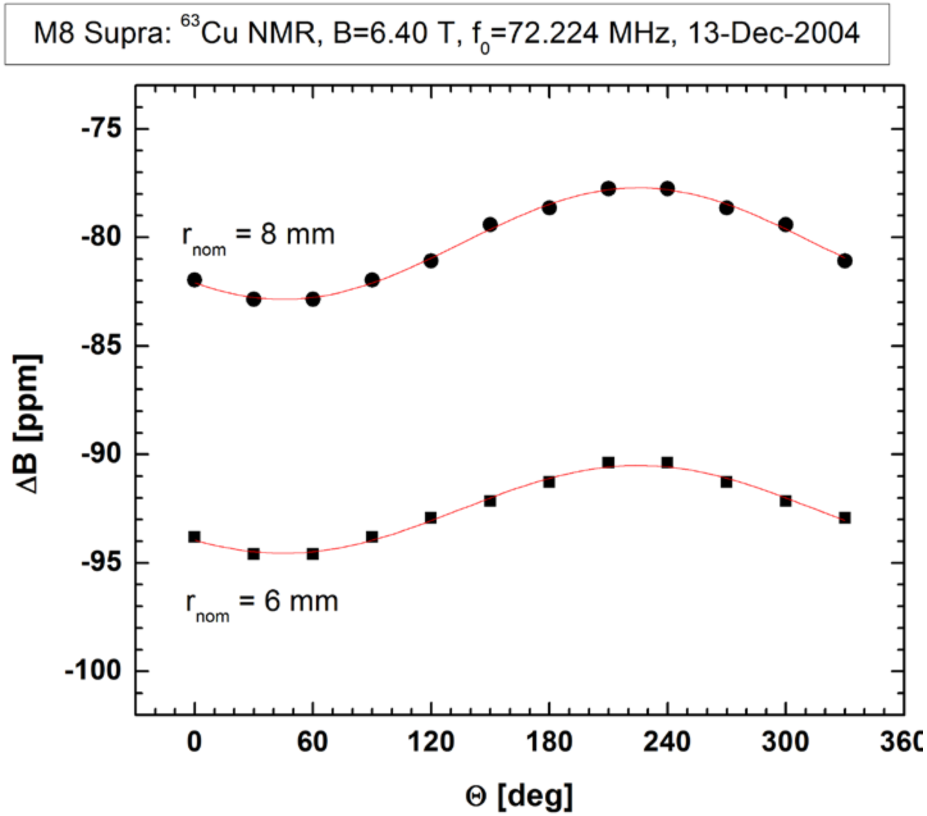


No variation for perfect centering!

Radial centering of axisymmetric magnets using NMR

Bitter magnet and superconducting outsert of hybrid magnet

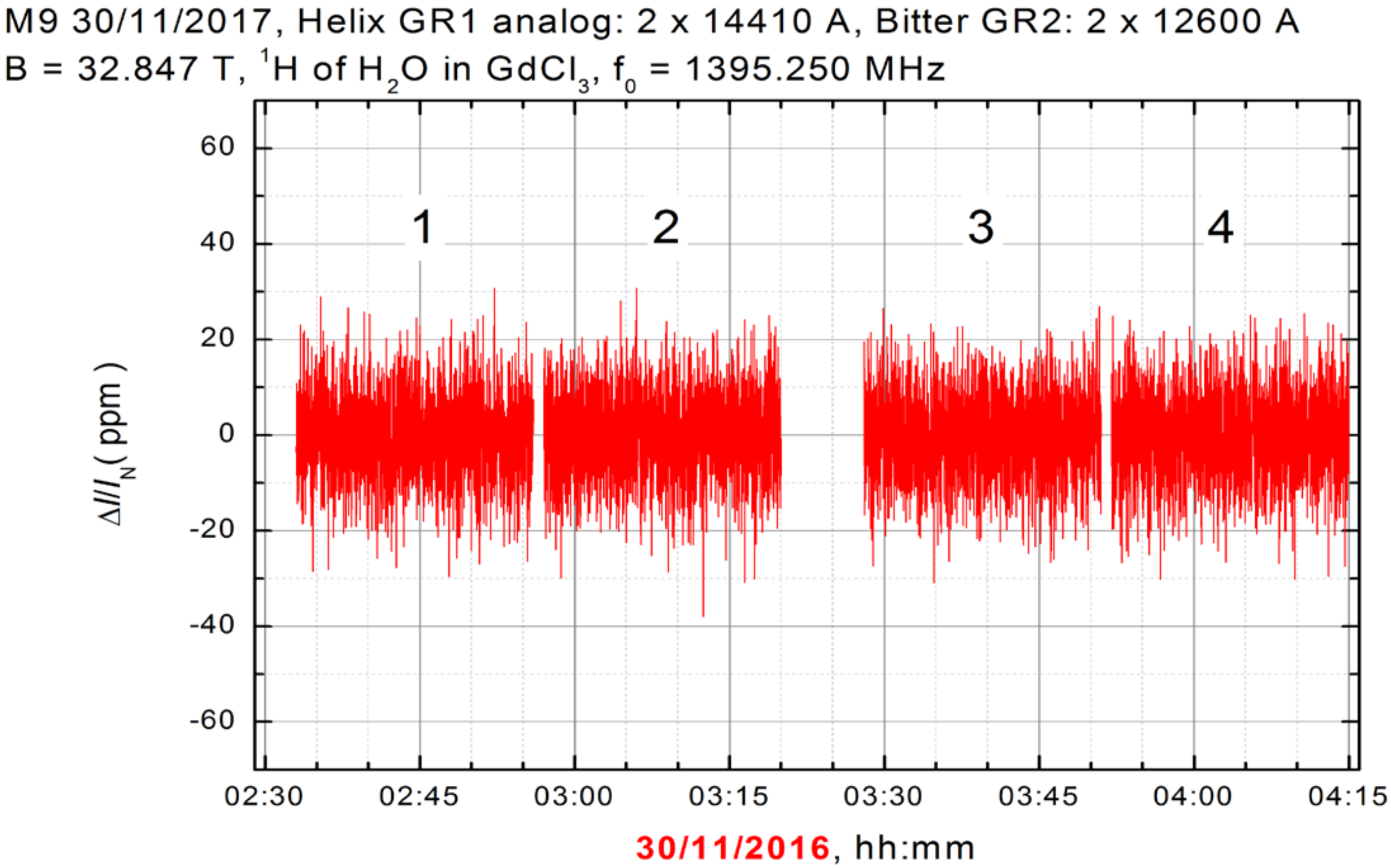




Bitter center at (x,y) = (-1 mm, 0 mm)

Supra center at (x,y) = (0.25 mm, 0.25 mm)

Example: NMR results during optimization period by Basis



Time record of 4 x 2000 NMR records, each taken during 20 minutes.

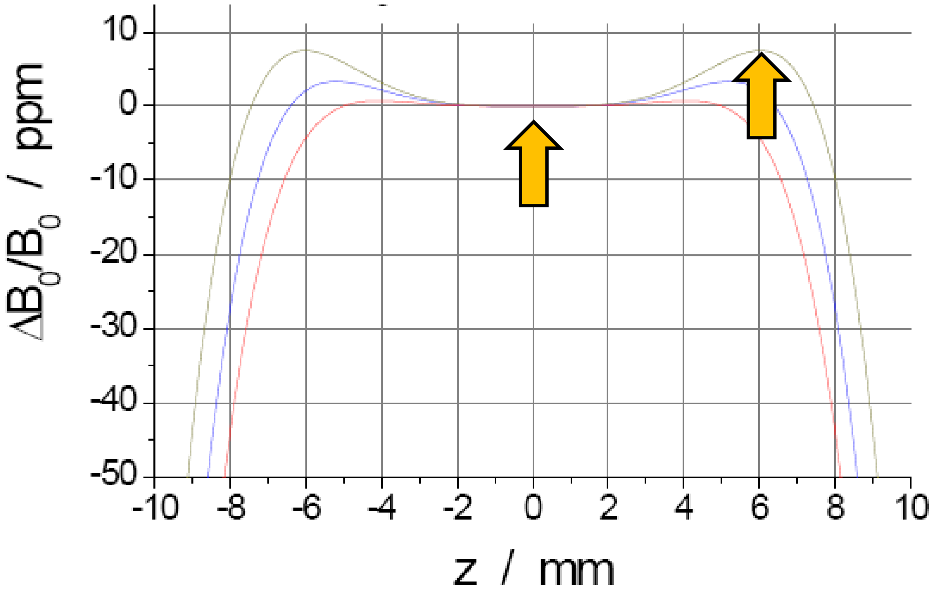
Normalization of field variation and subtraction of linear drift.

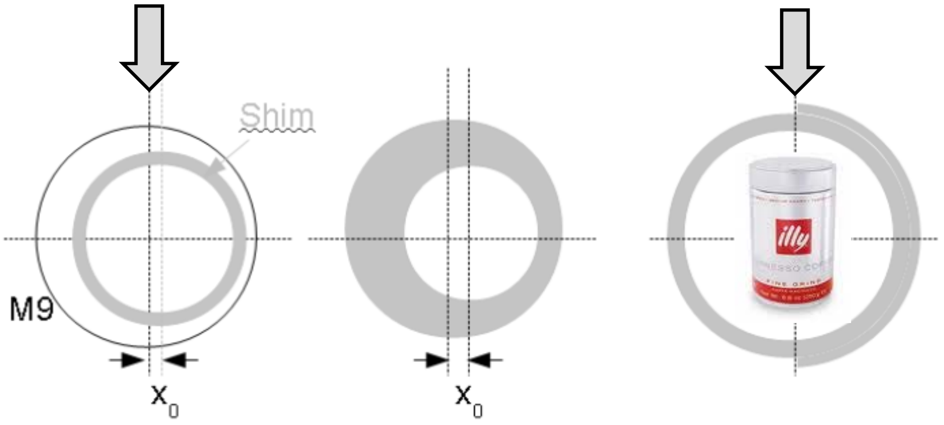
Analysis of data by counting of occurrences of variation values (= histogram) ….

Optimization and implementation of ferroshim at LNCMI

H. Stork

Second (smaller) region of enhanced homogeneity for spin lock placement along z-axis.

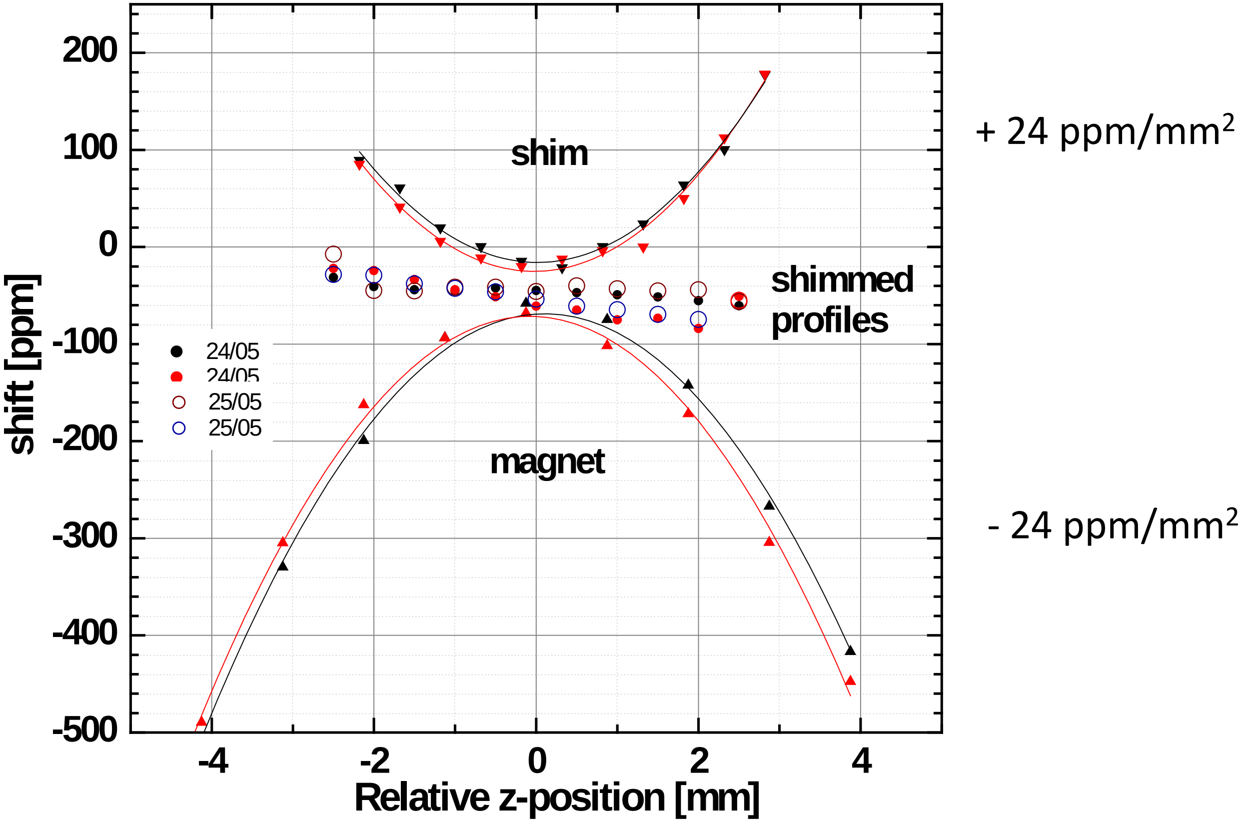




Options for radial gradient suppression.

Options for enhanced shim strength (10% more) by using Co/Fe alloy (Permendur)

Passive shimming performance at 29 T



✓ Effective reduction of axial and radial field inhomogeneity: $\Delta B/B$ ~ 20 ppm for Δz = 4 mm
Further optimization using liquid sample: $\Delta B/B$ ~ 10 ppm for Δz = 5 mm

➢ Importance of precise (sub-mm) 3d shim and sample positioning system.