RUHR-UNIVERSITÄT BOCHUM
FAKULTÄT FÜR PHYSIK UND ASTRONOMIE

RUB

PSTP 2013
INTERNATIONAL WORKSHOP
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Recent research activities and results of the Bochum/Bonn Polarized Target Group

G. Reicherz

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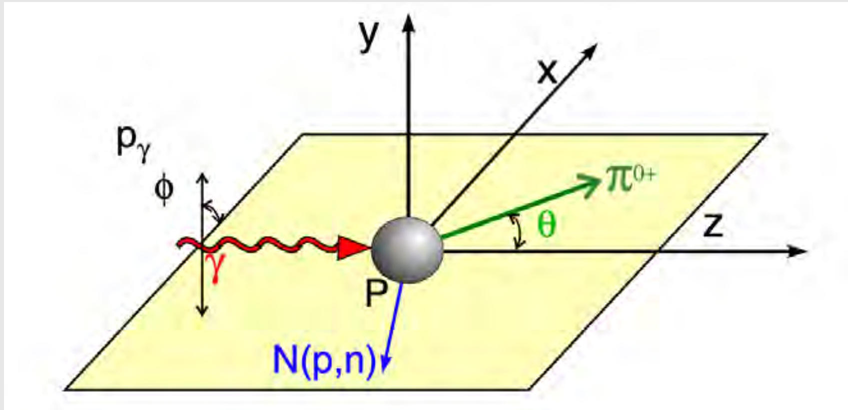
- Introduction
- Bonn Frozen Spin Target
- Internal magnet development
- t_{1e} measurements
- Material progress
- Bochum NMR box
- Summary

Introduction

Focus : Study of the nucleon resonance region (baryon spectroscopy) at ELSA by measuring single and double polarization observables

Polarized beam + polarized target + 4π detection system

Structure mapping at ELSA
- ➢ Model independent partial wave analysis
- ➢ Complete experiment

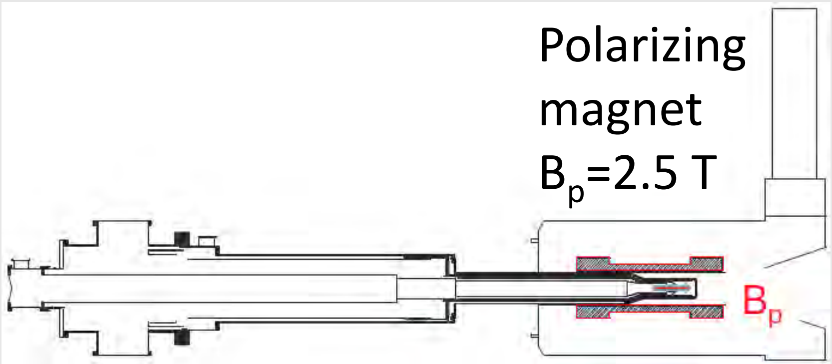


Photon		Target		
		x	y	z
unpolarized	σ	0	T	0
linearly	(-Σ)	H	(-P)	(-G)
circularly	0	F	0	(-E)

$$\begin{aligned}\frac{d\sigma}{d\Omega}(\Theta,\phi) = \frac{d\sigma}{d\Omega}(\Theta)\cdot\big[1 &- p_\gamma^{lin}\Sigma(\Theta)\cos(2\phi) \\ &+ p_t^x\cdot\left(-p_\gamma^{lin}H(\Theta)\sin(2\phi) + p_\gamma^{circ}F(\Theta)\right) \\ &- p_t^y\cdot\left(+p_\gamma^{lin}P(\Theta)\cos(2\phi) - T(\Theta)\right) \\ &- p_t^z\cdot\left(-p_\gamma^{lin}G(\Theta)\sin(2\phi) + p_\gamma^{circ}E(\Theta)\right)\big]\end{aligned}$$

Bonn Frozen Spin Target

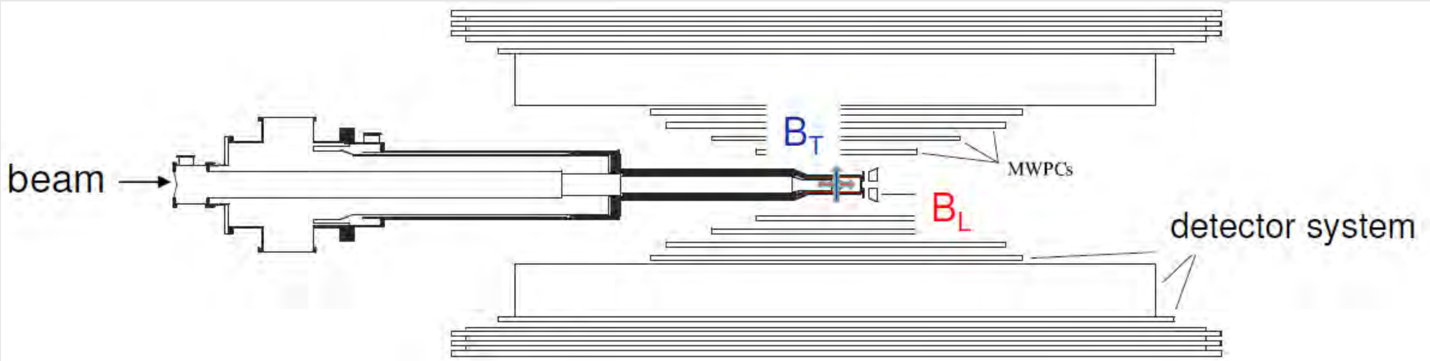
⇒ Frozen spin target + internal holding coil



Polarizing mode DNP
'continuous mode'
$P_{D/p} \sim 80 – 90$ %

Horizontal dilution refrigerator ($T \leq 50$ mK)
with internal 'longitudinal' or transversal
holding coil ($B_{T/L} \sim 0.5$ T -1 T)

Data taking (frozen spin) mode
$\tau \sim 300 – 1000$ h



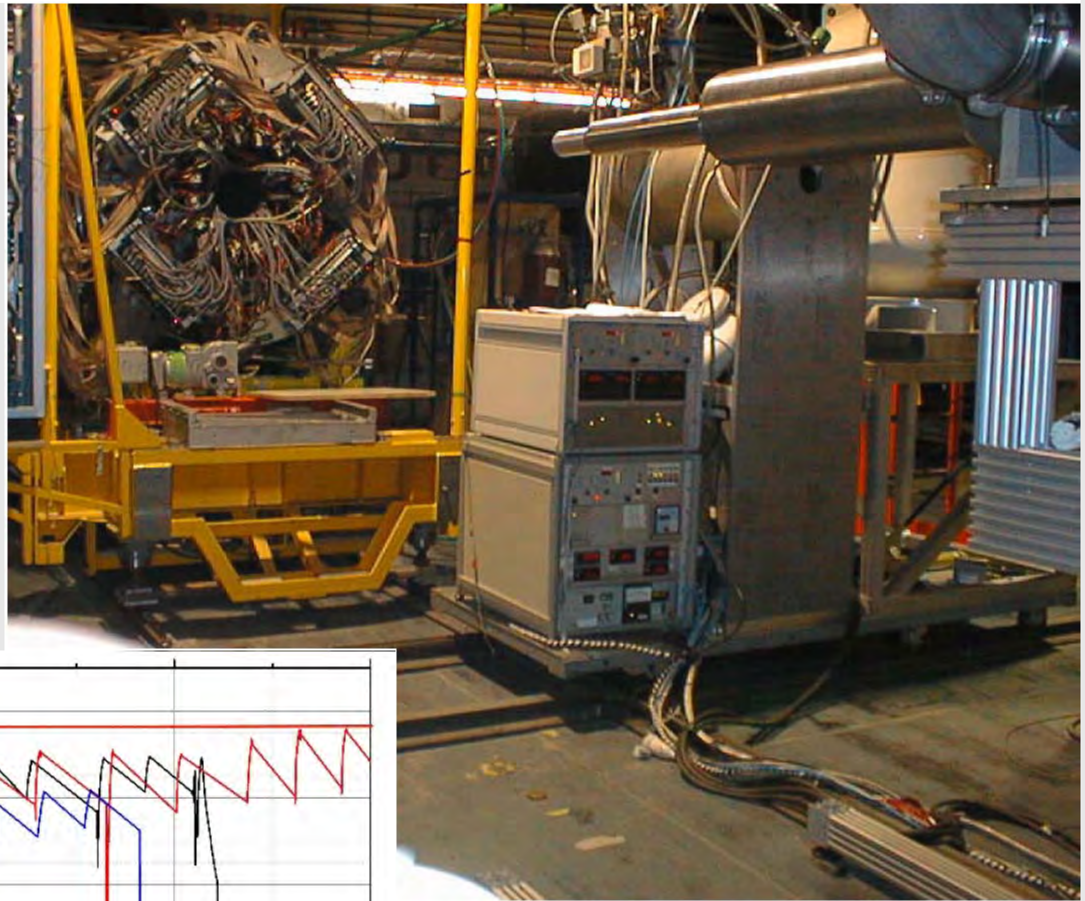
⇒ Large angular acceptance ($0.98*4\pi$) → complex handling (moving) system

Bonn Frozen Spin Target

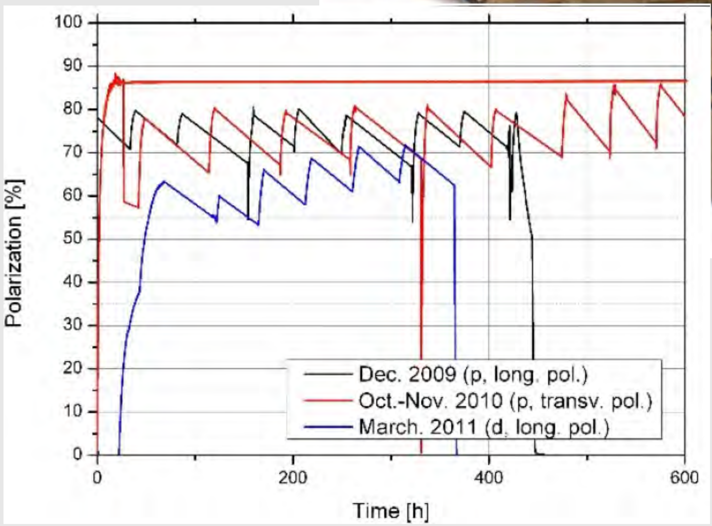
⇒ Frozen spin target + internal holding coil

Frozen spin technique limitations:

- Large acceptance target system requires dedicated railway system
- $FoM = \sigma n_T f^2 \bar{P}^2$



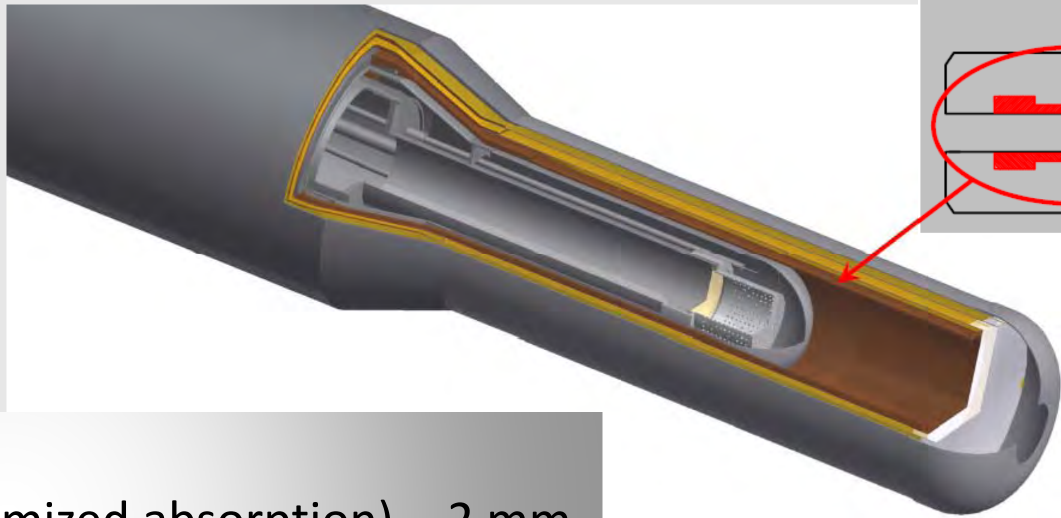
Combine advantage of the frozen spin technique with the advantage of a continuous polarization

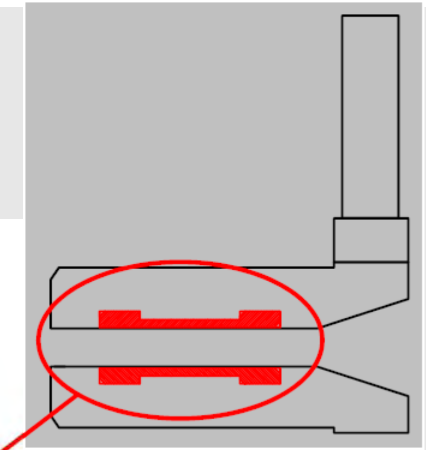


4π – continuous mode target with internal polarizing magnet

Internal magnet development

Idea: reduce the magnetic volume of the large external pol. magnet to the size/dimensions of the internal holding coil





- field strength: ~ 2.5 T
- as thin as possible (minimized absorption) ~ 2 mm
- homogeneity dB/B $\leq 10^{-3}$

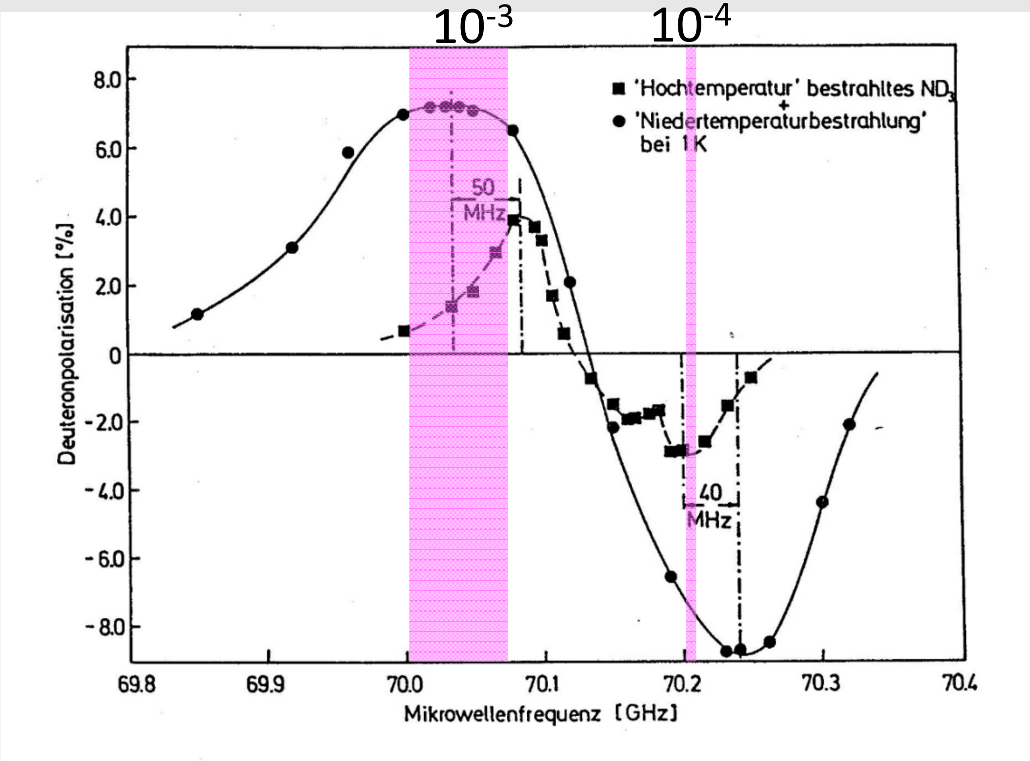
$$B = \mu_0 \cdot N \cdot \frac{I}{l}$$

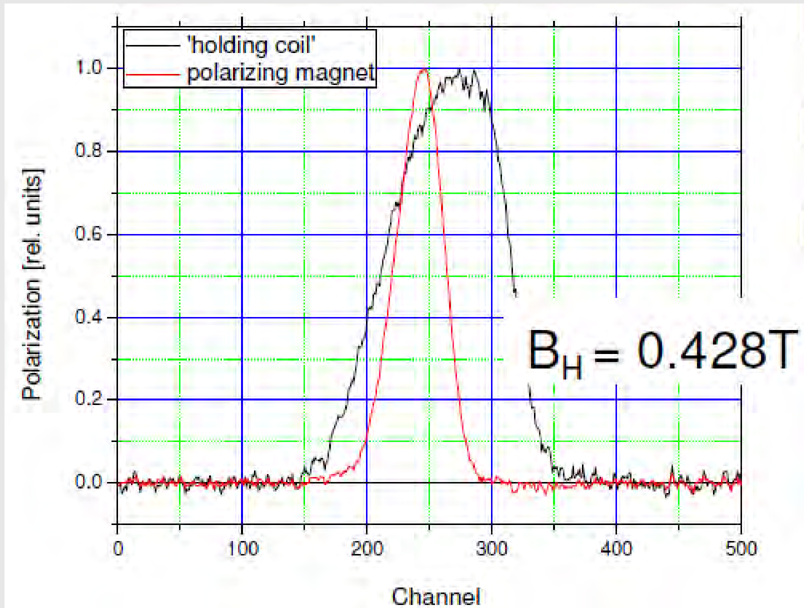
NC: ampere-turn : N·I ~ 300 kA → superconducting wire necessary
High current operation (~100 A) in a dilution refrigerator

Internal magnet development

Why do we need a homogeneity of $10^{-4} < \Delta B/B < 10^{-3}$

Depending on material and radical a homogeneity $< 10^{-3}$ is needed





NMR in a 4-layer holding coil (uncorrected solenoid $\Delta B/B \sim 10^{-3}$)

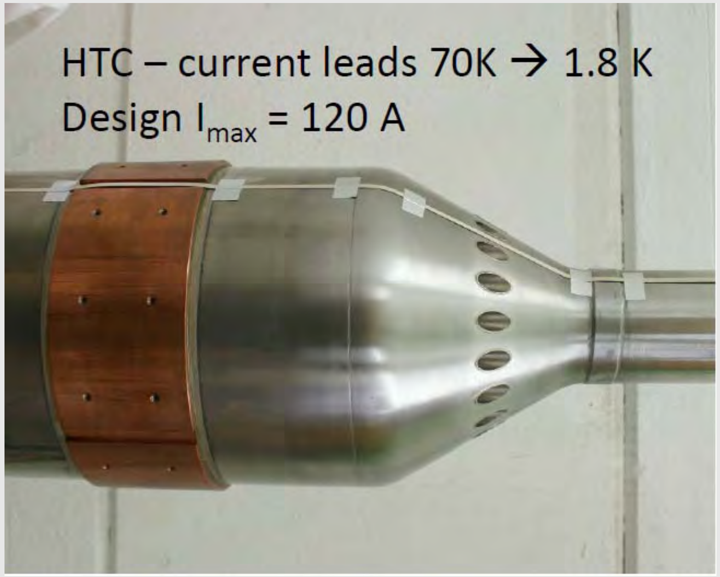
DNP requires separation of the nucleon spin levels

Low-T refrigerator with high current leads Bochum/Bonn

Horizontal dilution refrigerator incl. internal polarizing magnet opt. run as ‚frozen spin target‘

- A 'continuous mode' operating target with large angular acceptance (~4π).
- $T_{min} \approx 30$ mK, $T_{continuous} \approx 200$ mK @ 100 mW.
- High luminosity L $\sim 10^{33}/cm^2s$ (N $\approx 10^{10}/s$).
- High mean polarization.
- Equipped with an internal superconducting polarizing magnet for permanent DNP and high current leads.

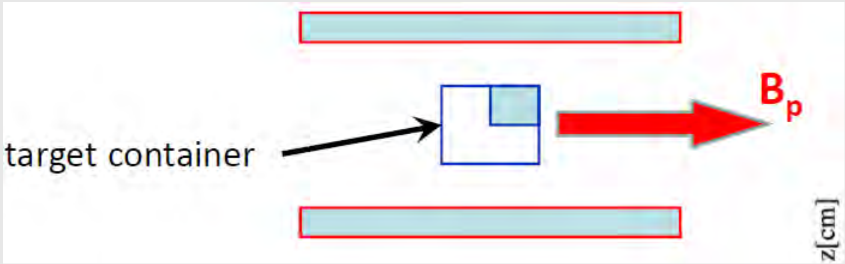
Low-T refrigerator with high current leads Bochum/Bonn



- Construction of the heat exchangers/components nearly completed
- Low temperature heat exchangers under construction at Dubna
- First cold test in short time

Design of low mass polarizing solenoid $10^{-4} < \Delta B/B < 10^{-3}$ for the new Bonn refrigerator

Fixed Parameters : length ~ 150 mm, ∅ 45 mm
total thickness < 2 mm
B_p = 2.5 T @ I_{max} < 100A



Uncorrected solenoid (B = 2.5T @ 90 A)

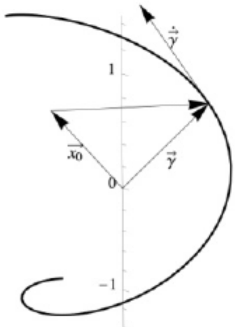
Biot-Savart

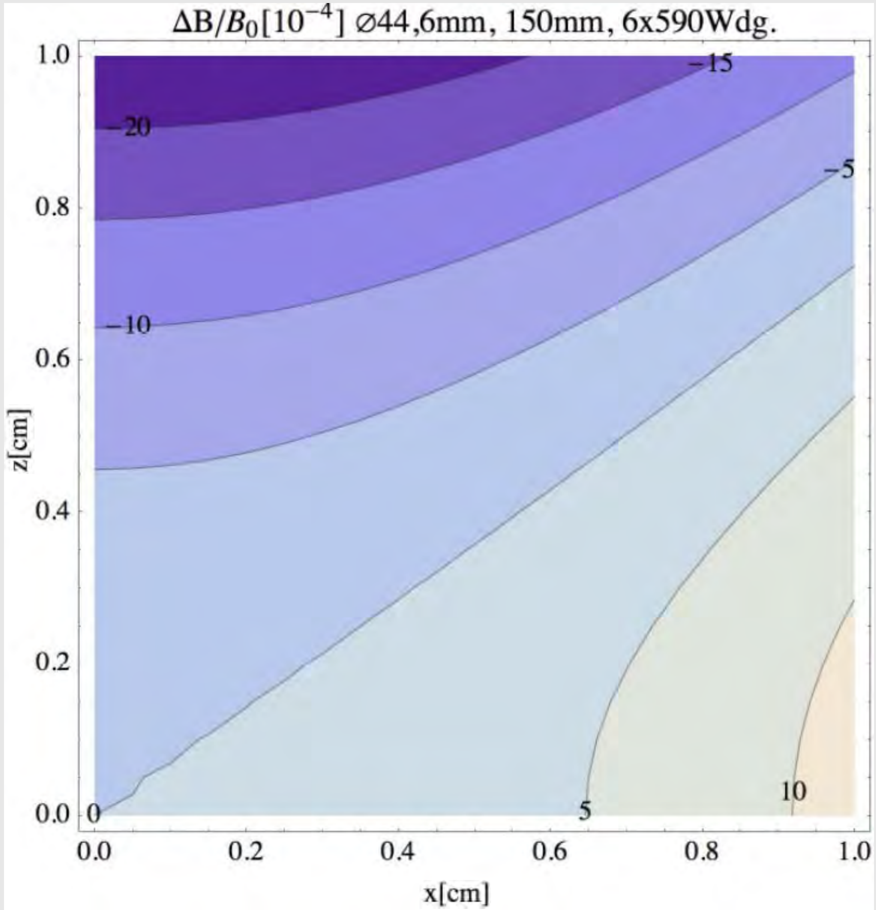
$$\vec{B}(\vec{x_0}) = \frac{\mu_0}{4\pi} I \int \frac{(\vec{\gamma}(t) - \vec{x_0}) \times \frac{\dot{\vec{\gamma}}(t)}{|\dot{\vec{\gamma}}(t)|}}{|(\vec{\gamma}(t) - \vec{x_0})|^3} dl$$

mit

$$dl = \sqrt{\dot{\vec{\gamma}}(t)\dot{\vec{\gamma}}(t)} dt$$

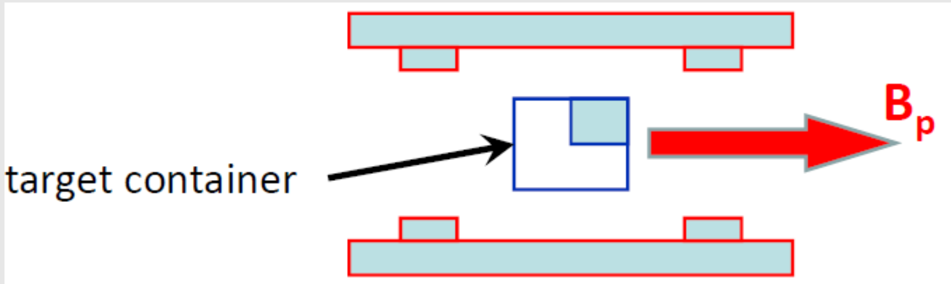
$$\vec{\gamma}(t) = (x(t), y(t), z(t))$$



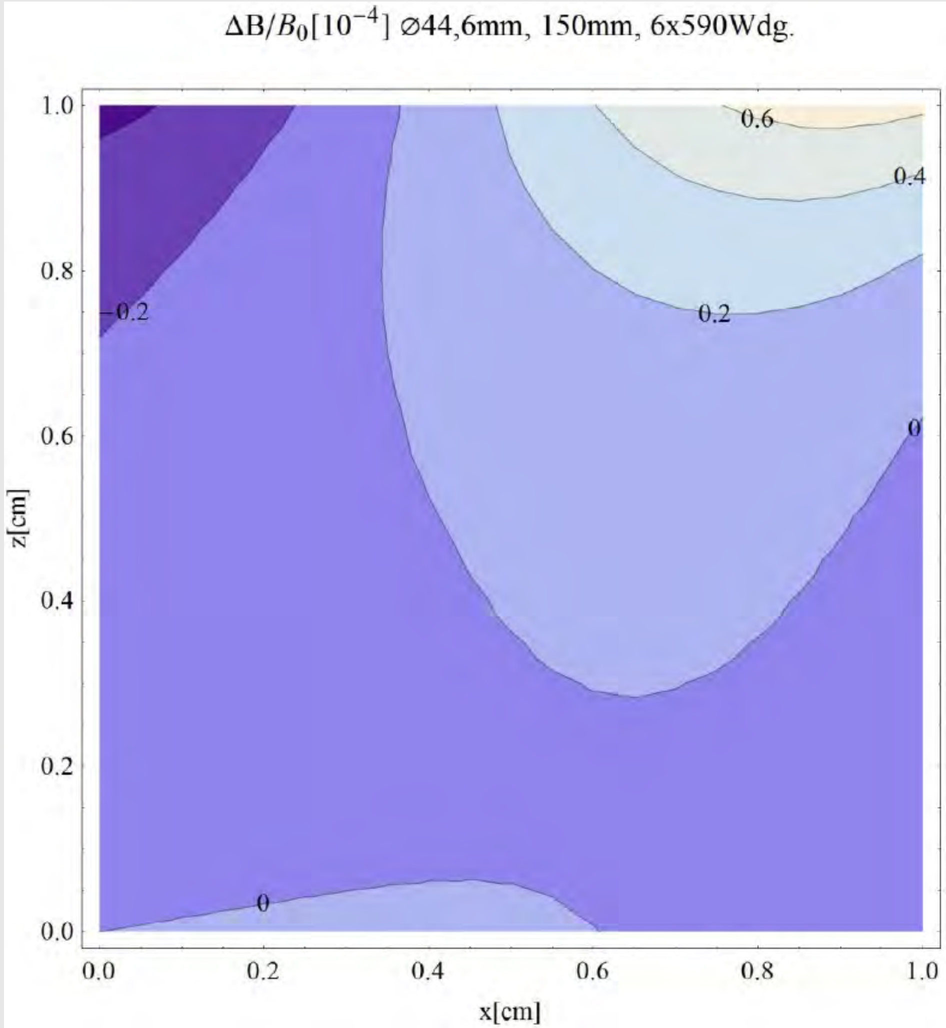


Design of low mass polarizing solenoid $10^{-4} < \Delta B/B < 10^{-3}$ for the new Bonn refrigerator

corrected solenoid : ‘inverse notched coil’,
B = 2.5T @ 90 A

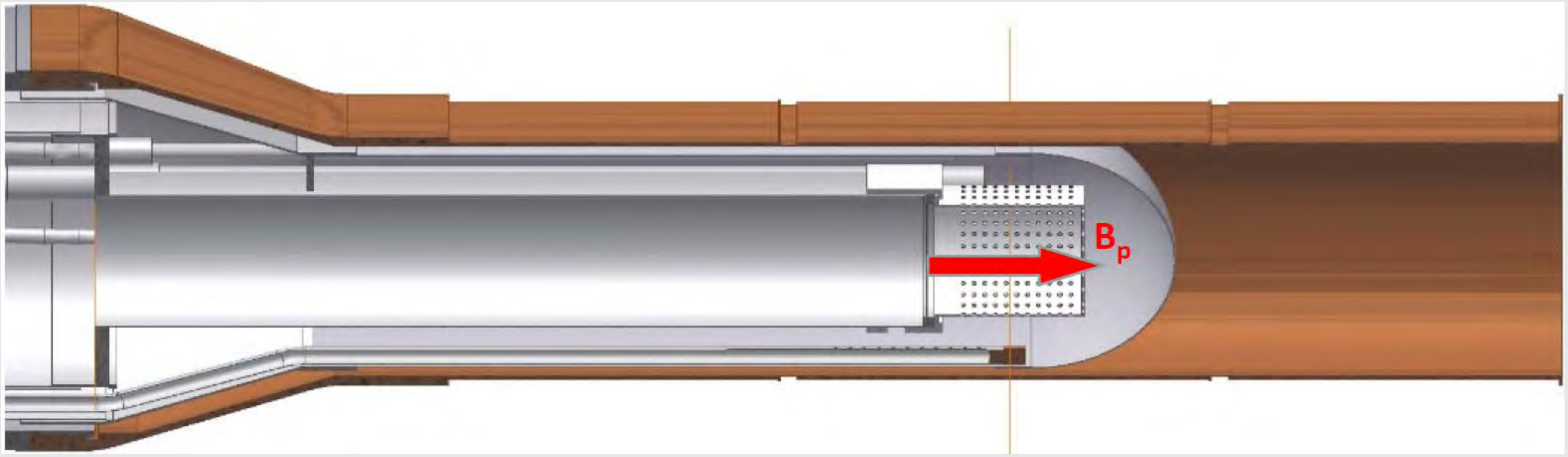


- $\Delta B/B < 10^{-4}$
- 6 layers, 0.25F54 1:1.35, 590 wdg.
- 2 corrector notches (2 · 8 windings)
- Thickness 1.8 mm
- High accuracy winding is mandatory



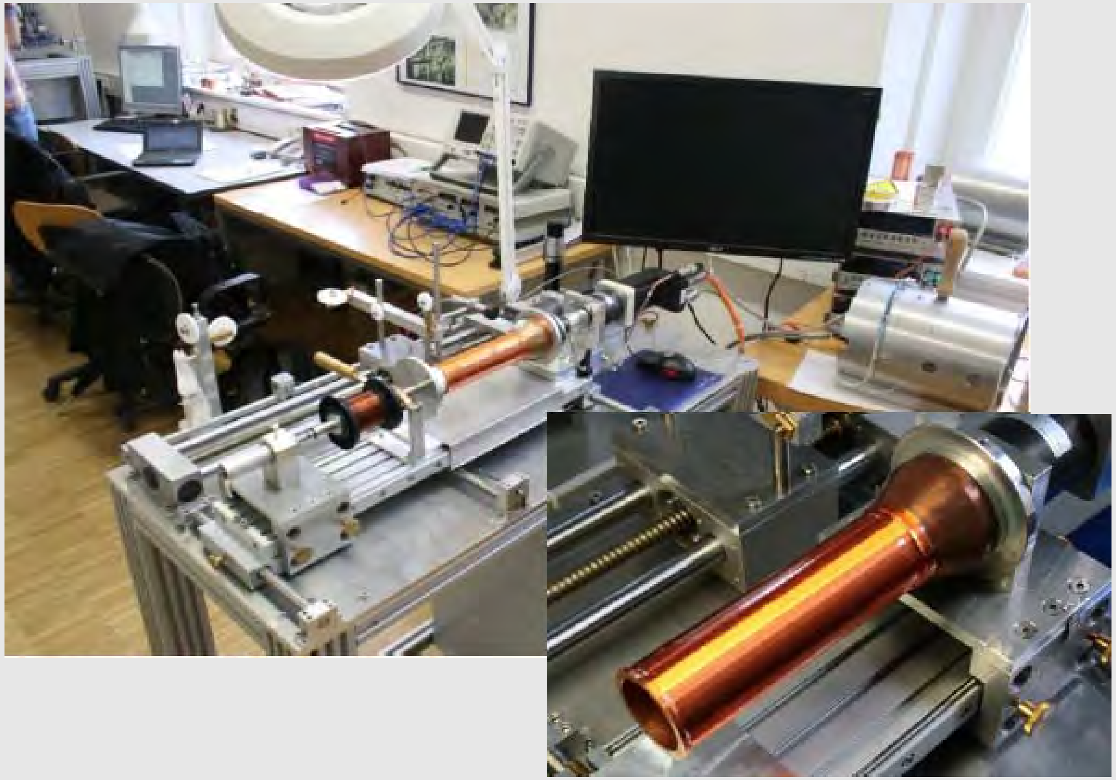
Design of low mass polarizing solenoid $10^{-4} < \Delta B/B < 10^{-3}$ for the new Bonn refrigerator

corrected solenoid : ‘inverse notched coil’, B = 2.5T @ 90 A for the new refrigerator



Prototype production / test of low mass super-conducting solenoid for high field Bonn / Mainz

First test coils have been wound in Mainz and Bonn



- High accuracy layer by layer wet winding has been solved (non standard or commercial technique)
- First test coils successfully tested in thermal cycles
- First cold test and field measurements at 4.2K have been done at Mainz / Bonn in short time

T_{1e} measurements

First moment

$$M_1^{IS} = \frac{1}{3}\mu_0 \langle \xi \rangle \gamma_I \gamma_S S \hbar n_S P_S$$

Shape factor of the sample

$$\langle \xi \rangle = \frac{1}{N_I}\sum_j \xi_j = \frac{1}{N_I}\sum_j \left[\frac{3}{4\pi\, n_S}\sum_\mu \frac{1-3\cos^2\theta_{j\mu}}{r_{j\mu}^3}\right]$$

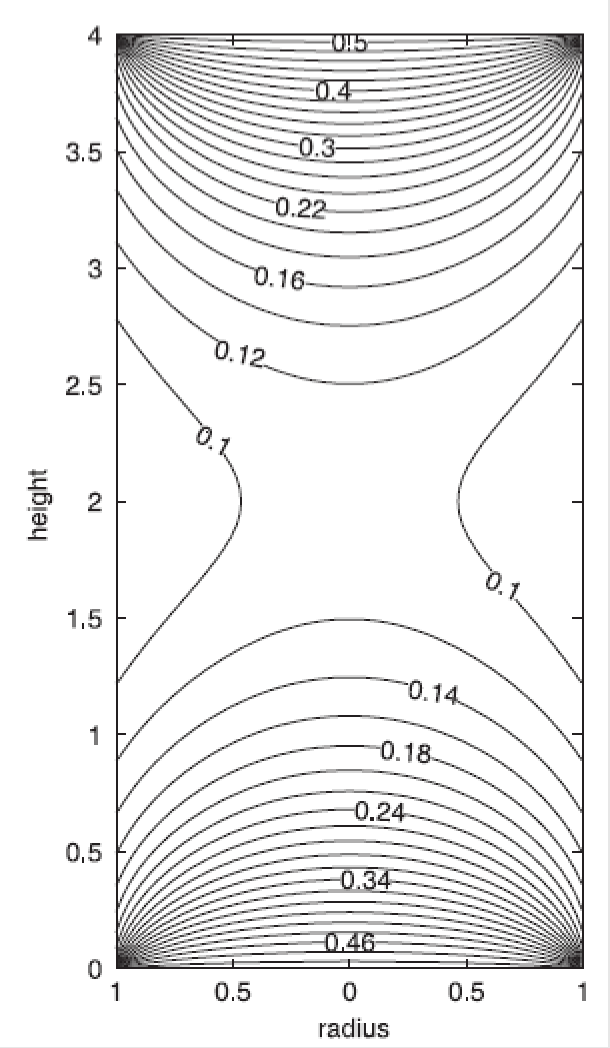
Demagnetization factor

$$\xi = 3D_{zz} - 1$$

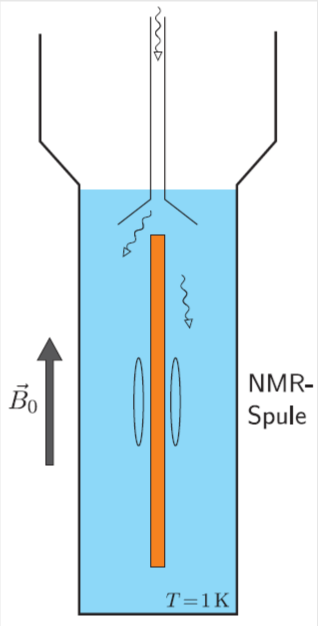
Frequency shift in hertz

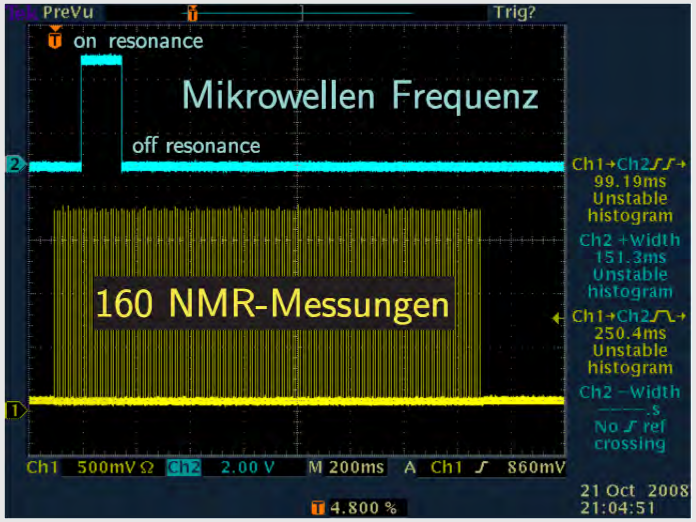
$$\Delta f = \frac{\mu_0}{6\pi}\langle \xi \rangle \gamma_I \gamma_S S \hbar n_S \cdot \left(1 - \frac{\tau_e}{T_{1e}}\right)\tanh\left(\frac{\gamma \hbar B}{2kT}\right)$$

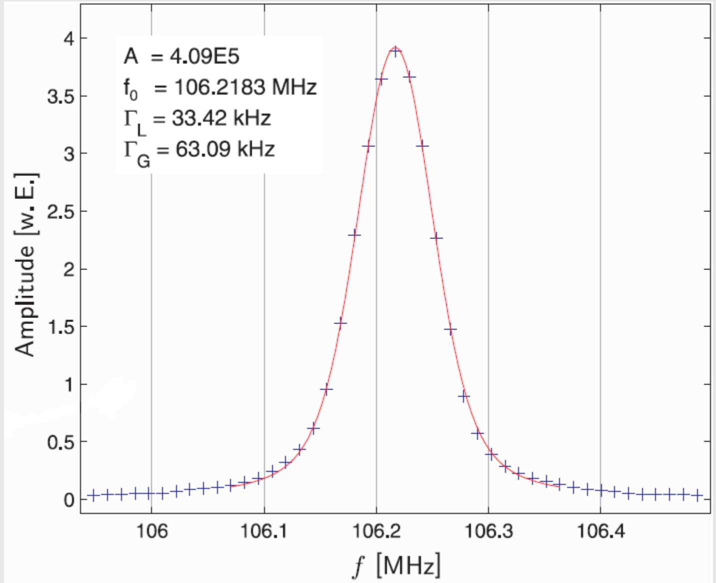
D_{zz} in a (h:d = 2:1) cylinder



T_{1e} measurements (NEDOR)

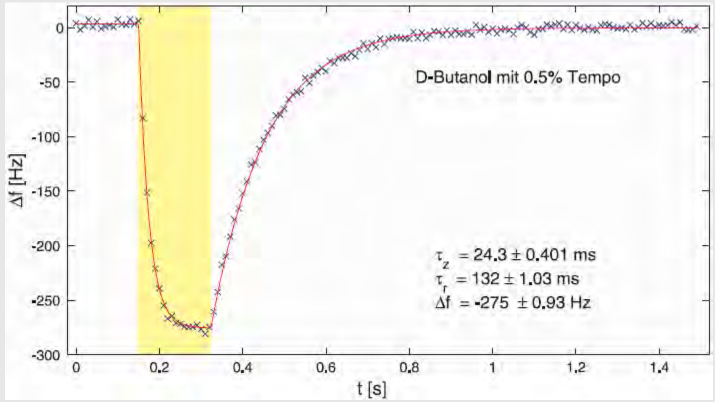






$$T_{1e}^{-1}(T) = \frac{1}{T_{1e}^{1K}} \cdot \exp\left(\frac{T - 1\mathrm{K}}{\vartheta}\right)$$

$$\langle \xi \rangle = -0.987$$



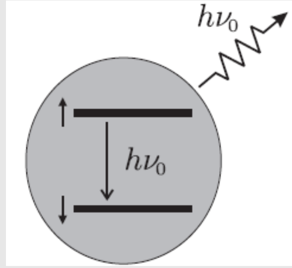
T_{1e} measurements

Frequency shift in Hz

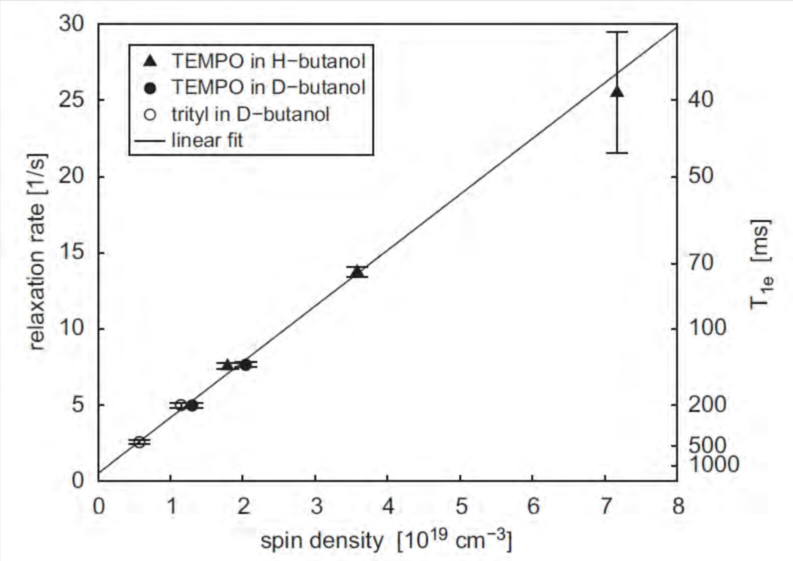
$$\Delta f = \frac{\mu_0}{6\pi}\langle\xi\rangle\gamma_I\gamma_S S\hbar n_S\cdot\left(1-\frac{\tau_e}{T_{1e}}\right)\tanh\left(\frac{\gamma\hbar B}{2kT}\right)$$

Relaxation via direct process

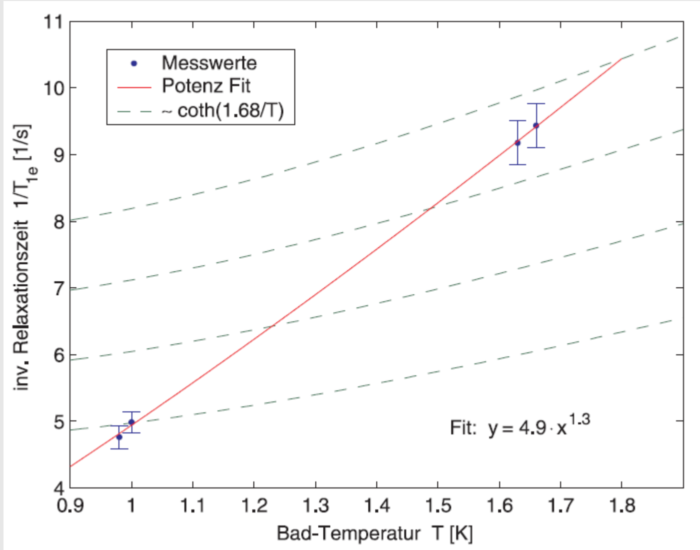
$$\frac{1}{T_1(\mathrm{d})} = \frac{24\pi^3\, h\, \nu_0^5}{\varrho\Delta_c^2\; v^5}\left|V^{(1)}\right|^2\coth\left(\frac{h\nu_0}{2kT}\right)$$



Paramagnetic relaxation rate



1/T_{1e} versus bath temperature

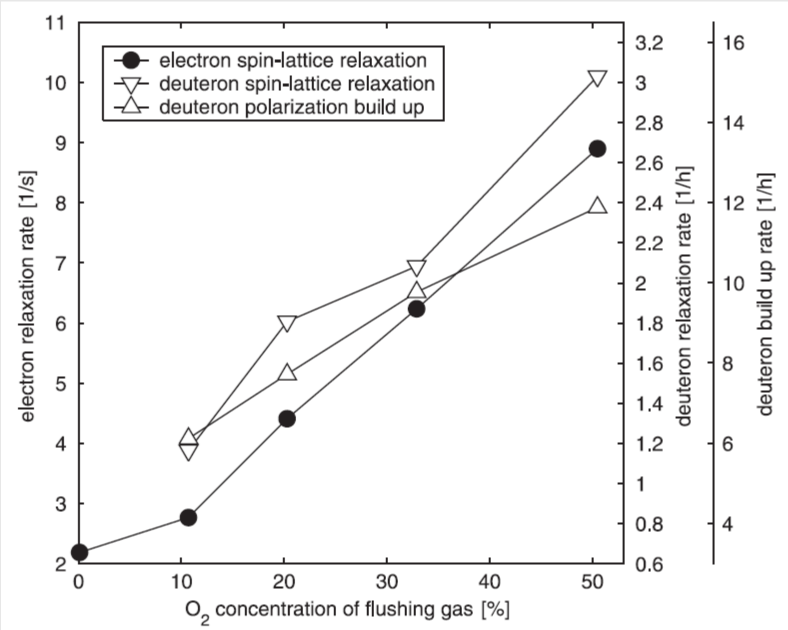


C. Hess| NIM A 694 (2012) 69–77

Evolution to lower temperatures?

T_{1e} measurements

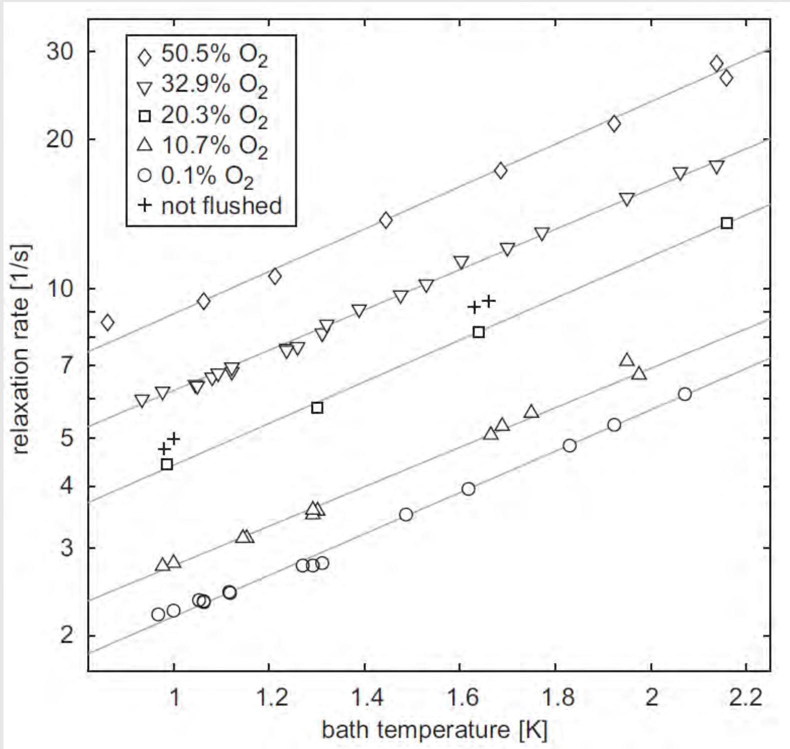
Rates of electron spin-lattice relaxation, deuteron spin-lattice relaxation and deuteron polarization build-up, as functions of the oxygen content.



Temperature dependence

$$T_{1e}^{-1}(T) = \frac{1}{T_{1e}^{1K}} \cdot \exp\left(\frac{T - 1K}{\vartheta}\right)$$

Fit function to extract T_{1e}



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T_{1e} via line-broadening

Reduced second moment

$$m_2^{IS} = \frac{(\mu_0 \gamma_I \gamma_S S \hbar n_S)^2}{9 N_I} \left[(1 - P_S^2) \sum_{j,\mu} \left(\frac{3}{4\pi n_S} \frac{1 - 3\cos^2\theta_{j\mu}}{r_{j\mu}^3} \right)^2 + P_S^2 \sum_j (\xi_j - \langle \xi \rangle)^2 \right]$$

Measurement:

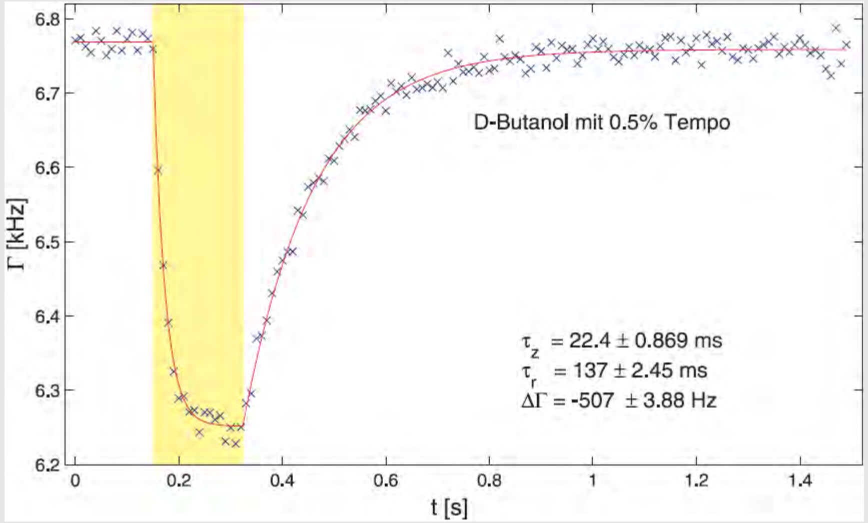
$$m_2 \propto P^2$$

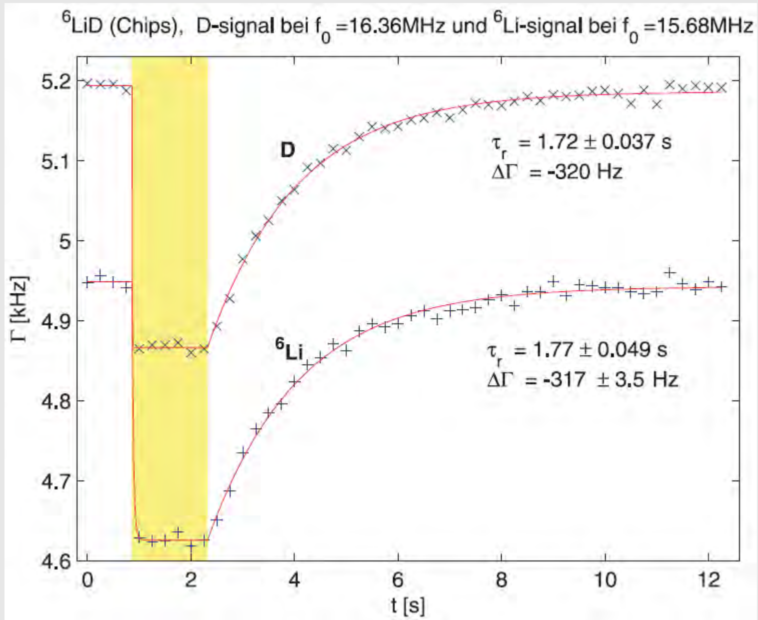
Model:

$$m_2 \propto (1 - P^2)$$

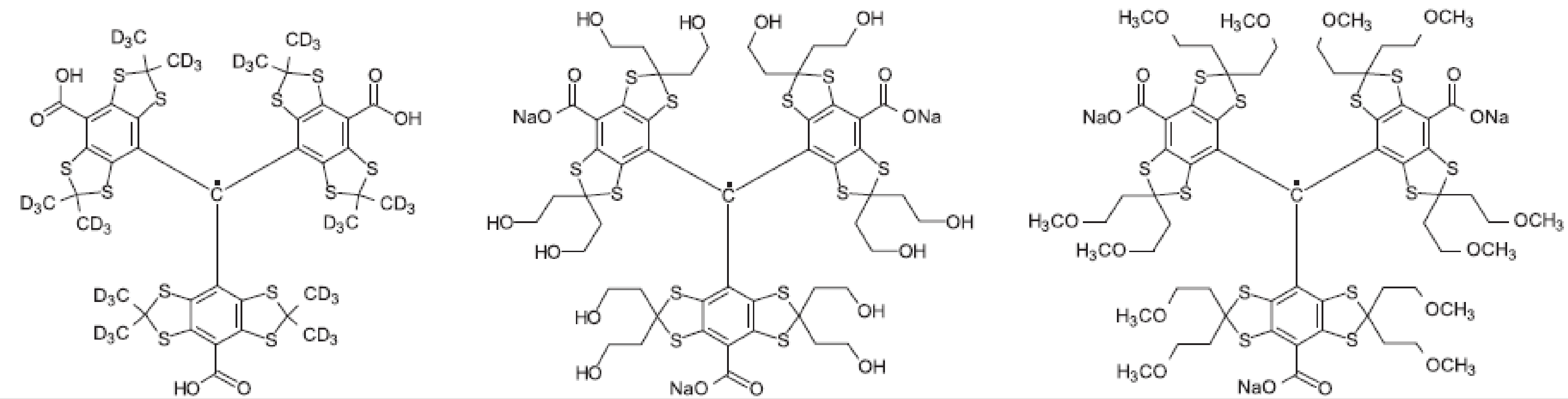
➢ Possible to measure T_{1e} from probes with any shape

^{6}LiD: $T_{1e} \cong 1.5$ to 1.8 s





The Trityl Radicals — Important progress for deuterated materials



Finland D36 (AH110355 deutero acid form) used for butanol-D10

Deuteron : up to 79% at 150mK/2.5T

Ox063 (AH100136 sodium salt) used for propandiol-D8

Deuteron: up to 81% at 150mK/2.5T

Ox063Me (AH 111 501 sodium salt) used for pyruvic acid

^{13}C: up to 74% at 900mK/5.0T

W. Meyer, et al., NIM A 631 (2011) 1-5

Important parameter: ESR line width and shape

- Zeeman Energy of a free electron

$$E_Z = -g_e \mu_B \vec{S} \cdot \vec{B}$$

- Contributions to the Electron Zeeman line width

$$\Delta E_{tot} = \underbrace{\mu_B(\vec{S} \cdot \hat{g} \cdot \vec{B}) + (\vec{S} \cdot A \cdot \vec{I})}_{in\,\mathrm{hom}} + \underbrace{E_D}_{\mathrm{hom}}$$

Hom. → dipol-dipol interaction → between electrons

Inhom. → hyperfine interaction → magnetic nuclei → indep. of B_0

Inhom. → g-factor anisotropy → crystal field → dep. of B_0

- Try to minimize the energy spread ΔE_{tot}
  - Find a suitable doping method → $\Delta E_{HFS} \sim \Delta E_D$
  - Try radiation doping if only low μ nuclei present

Bochum measurement

D-Butanol	EDBA	5.98 ± 0.03	12.30 ± 0.20	26
D-Butanol	TEMPO	3.61 ± 0.13	5.25 ± 0.15	34
D-Butanol	Porphyrexide	4.01 ± 0.15	5.20 ± 0.23	32
$^{14}ND_3$	$^{14}\dot{N}D_2$	$\approx 2 \ldots 3$	4.80 ± 0.20	44
$^{15}ND_3$	$^{15}\dot{N}D_2$	$\approx 2 \ldots 3$	3.95 ± 0.15	-
D-Butanol	Hydroxyalkyl	1.25 ± 0.04	3.10 ± 0.20	55
^{6}LiD	F-center	0.0	1.80 ± 0.01	57
D-Butanol	Finland D36	0.50 ± 0.01	1.28 ± 0.03	79
D-Propandiol	Finland H36	0.47 ± 0.01	0.97 ± 0.04	-
D-Propandiol	OX063	0.28 ± 0.01	0.86 ± 0.03	81

J. Heckmann, et al., Phys. Rev. B 74 (2006) 134418.

Result: The smaller the EPR line width, the higher the deuteron polarization value

Development of Polymer Targets

Advantages of polymer targets:

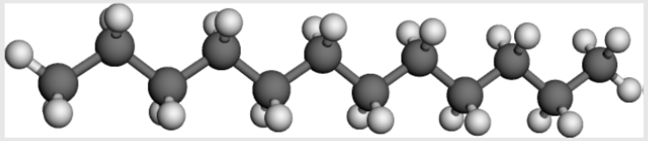
- Samples can be formed to practically any geometry.
- Handling of the samples is relatively safe.
- Samples contain only C and H (D) atoms making the background subtraction more precise.
- The dilution factor e.g. of polypropylene is slightly higher than that of butanol.

- D-Polyethylene CD_2: Paramagnetic centers by irradiation
  D.G. Crabb, Nucl. Instr. and Meth. A 526,56 (2004)
  35 % at 6.5 T/1K

- D-Polystyrene C_8D_8 : Paramagnetic centers by chemically doping with TEMPO
  B. van den Brandt et al., Nucl. Instr. and Meth., A536, 53 (2004)
  40 % at 2.5 T/100mK

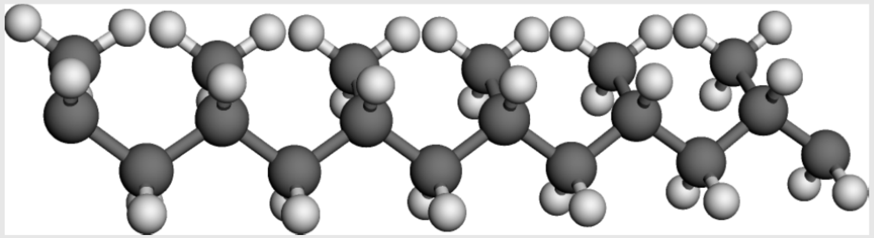
Development of Polymer Targets

Polyethylene: $(CH_2)_n$



- Can be classified by density: LLDPE, LDPE, HDPE

Polypropylene: $(CH_2 - CH - CH_3)_m$



- Can be classified by molecular weight M_w PP12, PP250, PP580.
- Initial choice of material was isotactic polypropylene.

Doping of materials : irradiation cryostats

Argon cooled cryostat

Wide temperature range cryostat

- Irradiation of materials with an e-beam creates structural defects for DNP e.g. NH_3, LiD.
- Regulated cooling is essential as beam is additional heat input.
- Materials can be irradiated in argon at 87 K.
- Materials can be irradiated in helium at an optimized temperature $90\ K < T < 270\ K$.

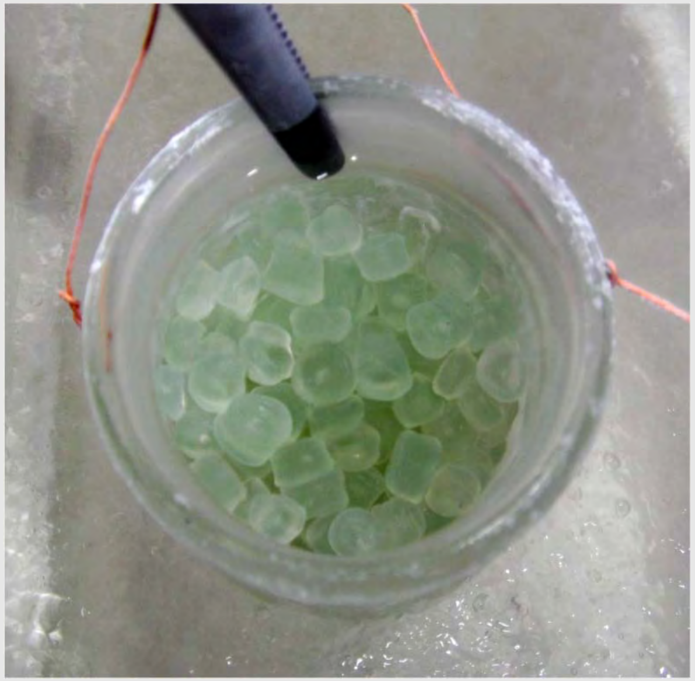
Doping of materials: LINAC1

pulse period	τ_{pulse}	1.2 µs
maximum current	I_{max}	250 mA
electron end energy	E_e	20 MeV
repetition rate	ν_{rep}	50 Hz

mean current	I	15 µA
beam power	P_{beam}	300 W
power deposited	P_{sample}	(30-45) W

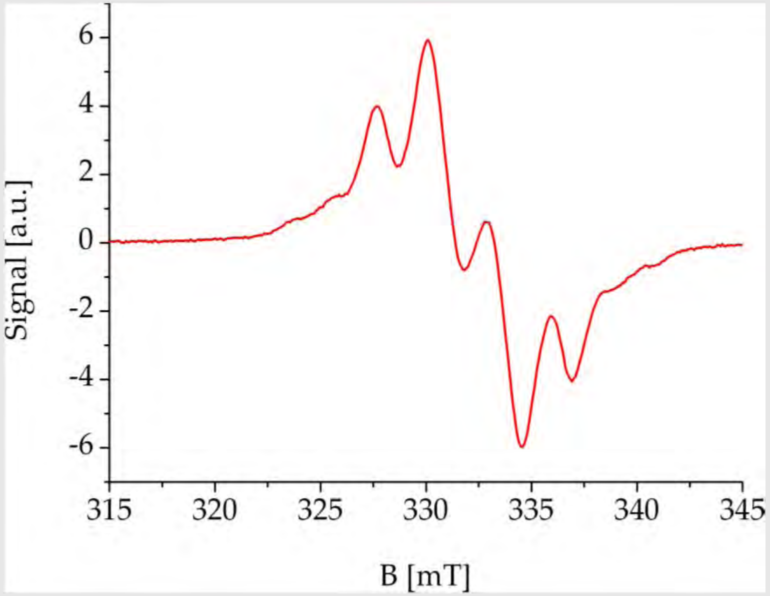
- A "standard" dose of $10^{17}\ \frac{e^-}{cm^2}$ creates $10^{19}\ \frac{e^-}{g}$ in anorganic materials.
- Typical irradiation times of anorganic materials in the region of hours.
- Organic materials are more susceptable to radiation damage and shorter irradiation times are sufficient.

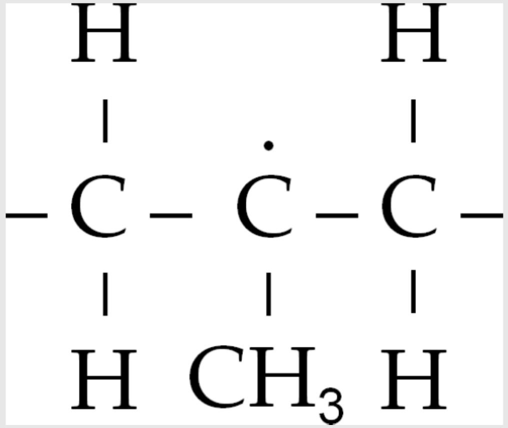
Development of Polymer targets radiation doping



- Initial calculation was made for the needed dose: Estimated dose of 10^{14} e^-/cm^2 is needed.
- Samples irradiated with 20 MeV e^- in argon cryostat at LINAC1 at ELSA.
- Initial irradiation of material approx. 10 min : Charge injection of 4.19 mC.
- Radical concentration determined by ESR measurement confirms dose is in correct order of magnitude.

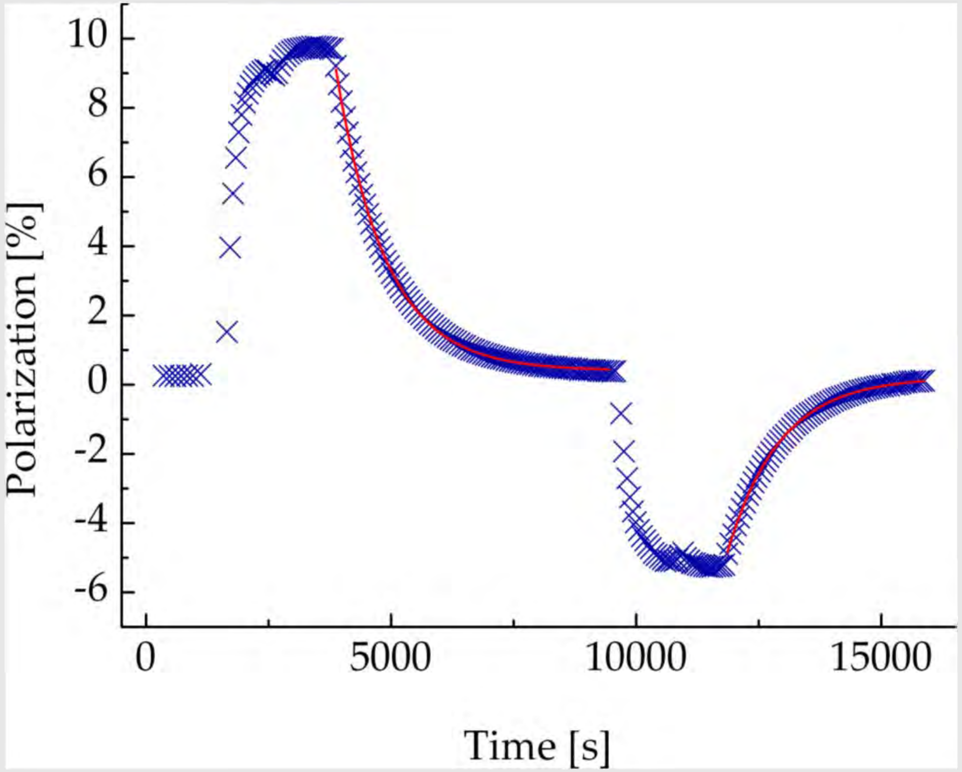
Development of Polymer Targets: EPR





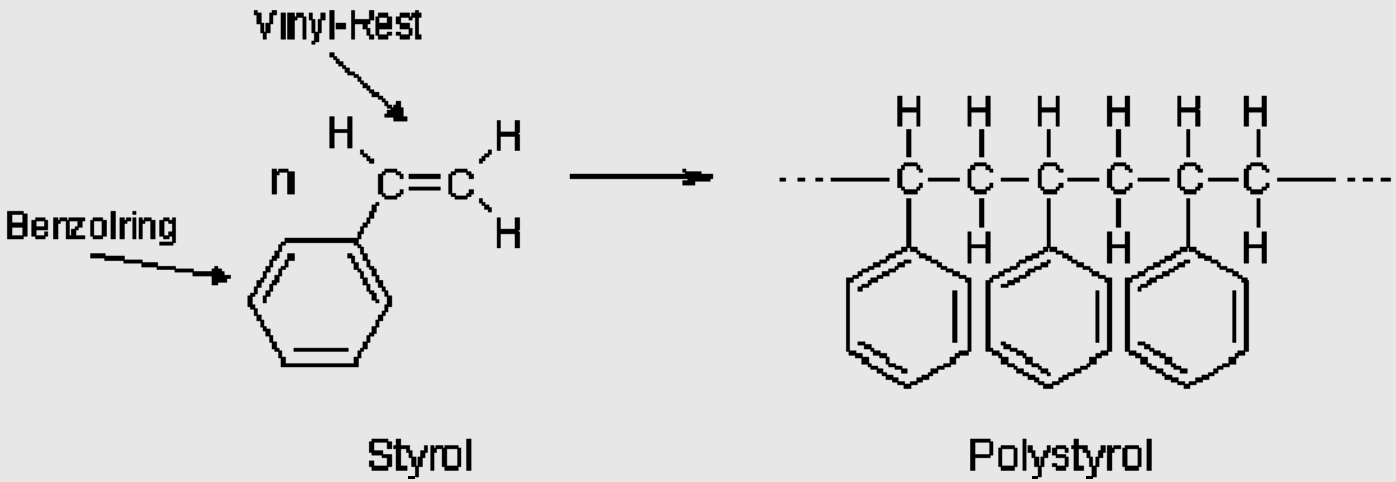
- The number of peaks in the ESR spectrum is given by $(2n_1I_1 + 1)$ $(2n_2I_2 + 1)$…
- 8 lines in spectrum are consistent with the interaction of 7 equivalent protons.
- Radical concentration of PP samples 5 x 10^{19} e^-/g.
- Samples show slight differences but not significant.

Development of Polymer targets: DNP

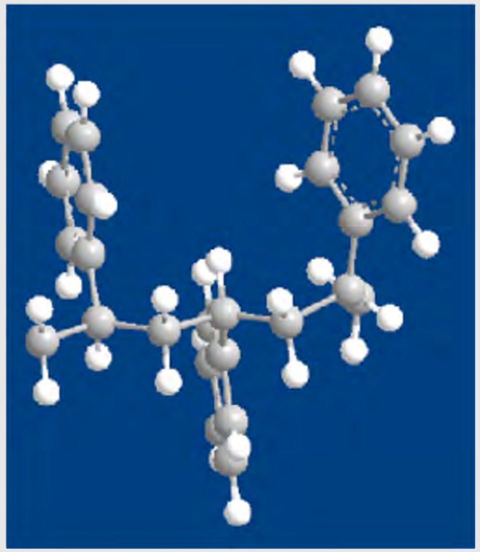


- Initial polarization measurements at 1 K gave $P_{max} \approx$ 4% and relaxation rates 1 min. Sample relaxation was too fast to build up a higher polarization.
- With subsequent annealing of sample it was possible to reach $P_{max} \approx$ 10% and $\tau_{heated} \approx$ 1h . Very promising result considering that the radical concentration may be too high.

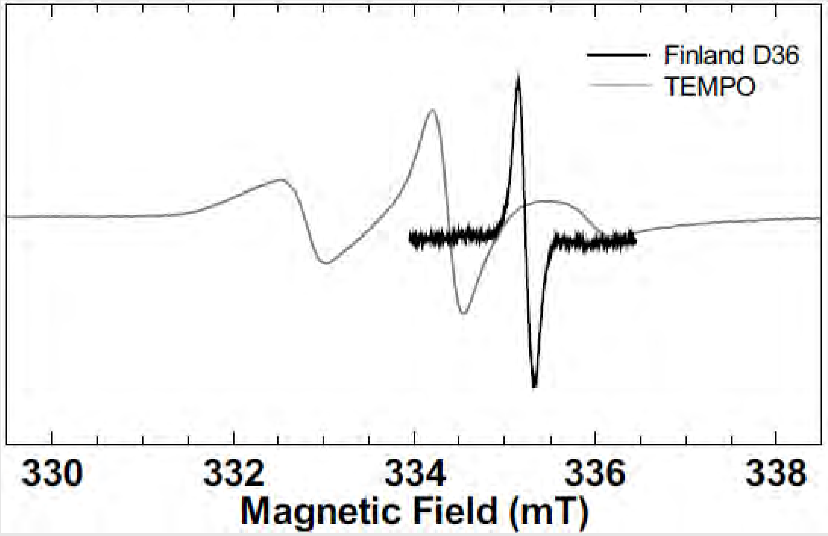
Polymer deuterated Polystyrol (~styrene) PS

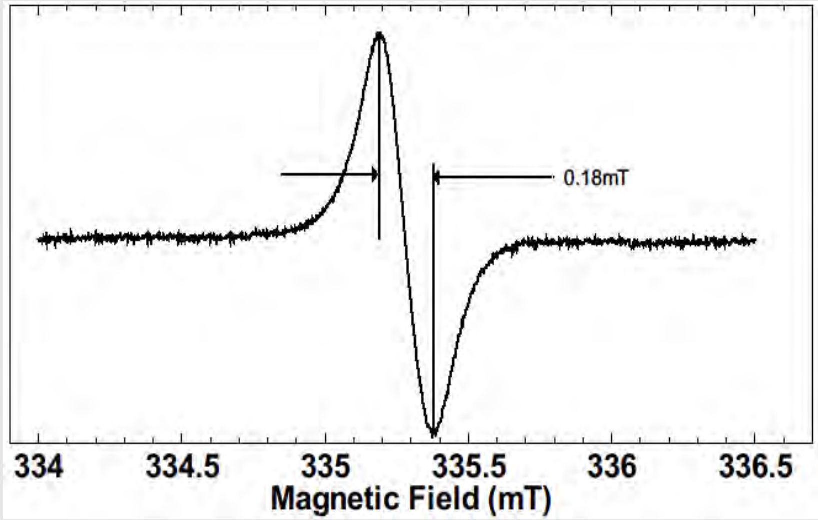


C_8D_8 dilution factor 0.143 (D-Butanol : 0.238)
Same advantages like the other Polymers



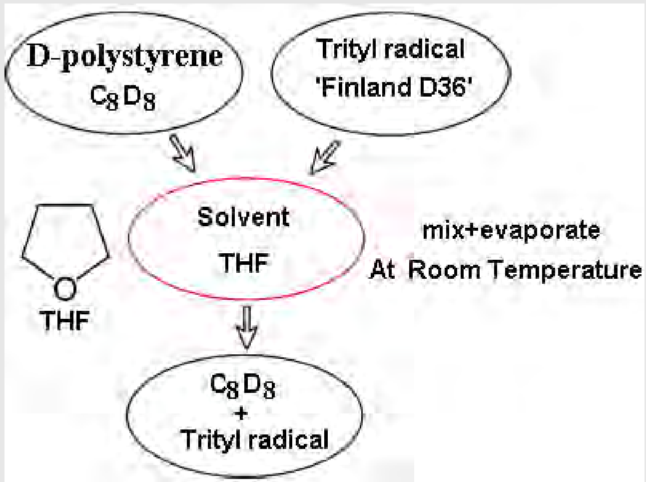
Preparation of trityl radical in D-Polystyrene





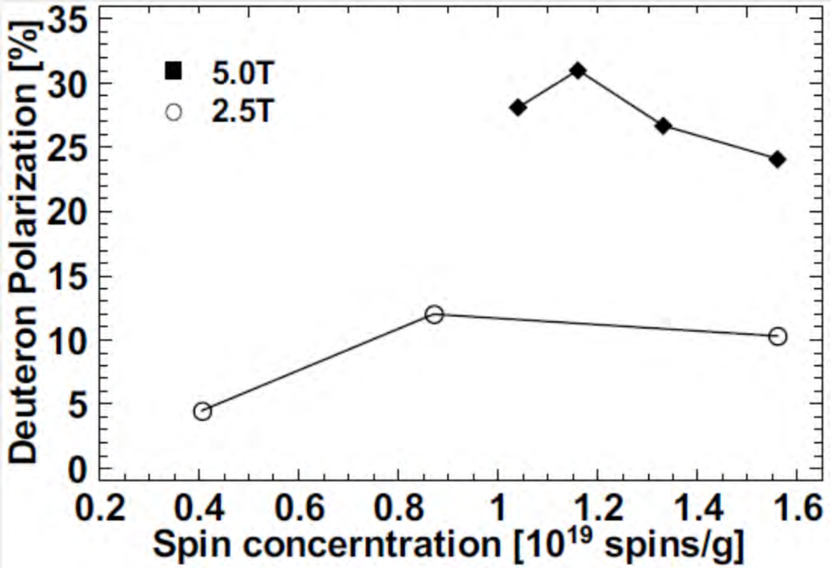
***g*-factor anisotropy:** $\Delta g / g \approx 3.0 \cdot 10^{-4}$

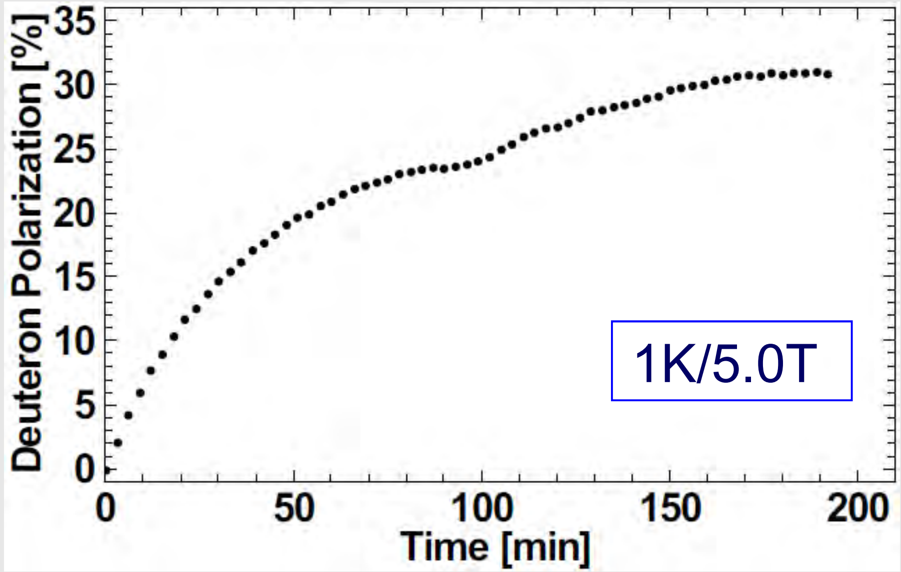
- Doping C_8D_8 with Finland D36



Homogenous and transparent foil (70µm)

Polarization of Finland D36-doped C_8D_8





Spin conc. (spins/g)	Mag. Field (T)	$T_{build-up}$ (min)	$T_{1,d}$ (min)	Microwave Freq. (GHz)	d-pol. (%)	f^+-f^- (MHz)
0.87×10^{19}	2.5	76	80(T=1.01K)	69.877	+10.2	56
				69.933	-12.5	
1.16×10^{19}	5.0	47	139(T=0.99K)	139.736	+29.5	92
				139.828	-31.0	

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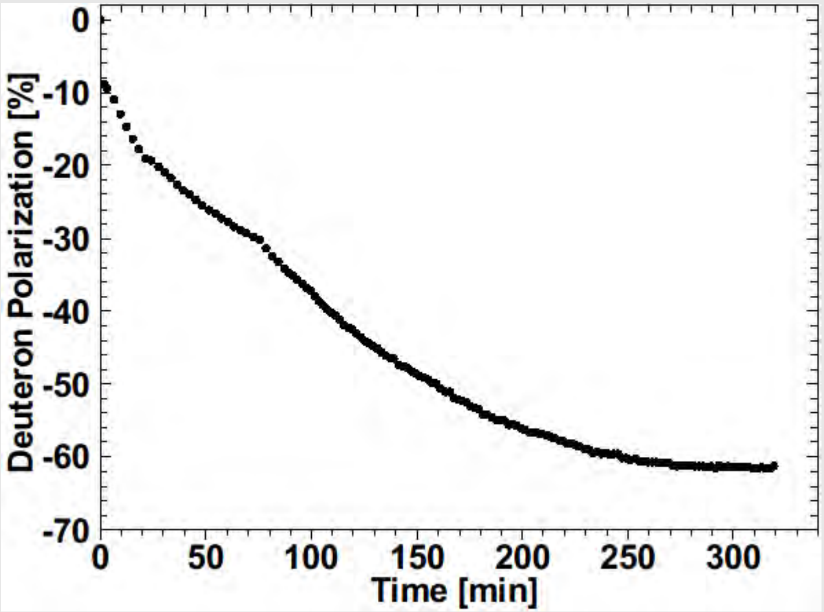
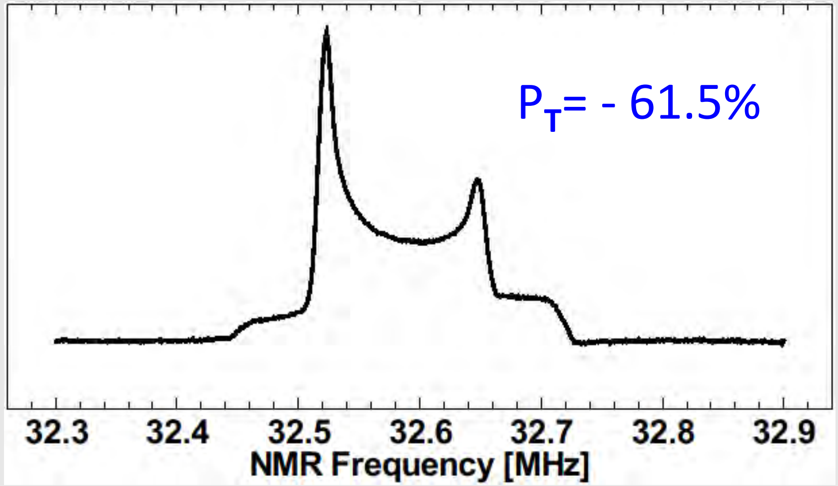
Polarization of Finland D36-doped C_8D_8





Sample	MW (GHz)	d-pola. (%)	$T_{1,d}$ (min)	$T_{build-up}$ (min)
d-PS(98%-d)	139.723	+56.1	863	100
+Finland D36	139.825	-61.5		

$f_{d,NMR}$=32.6MHz

Temperature = 400 mK and magnetic field = 5 T

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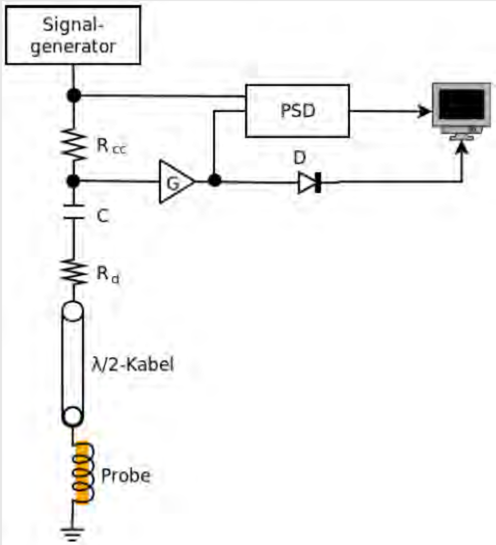
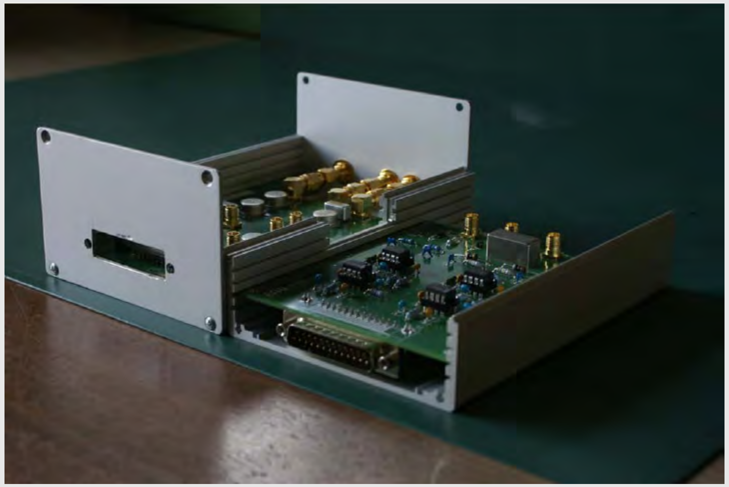
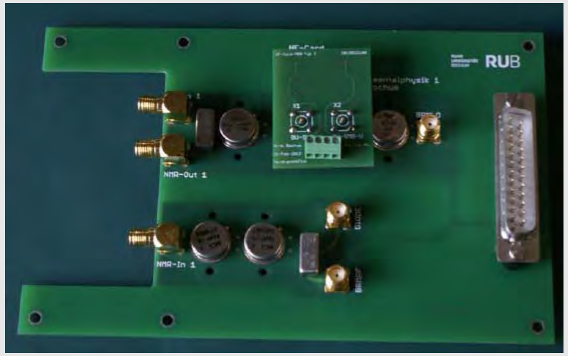
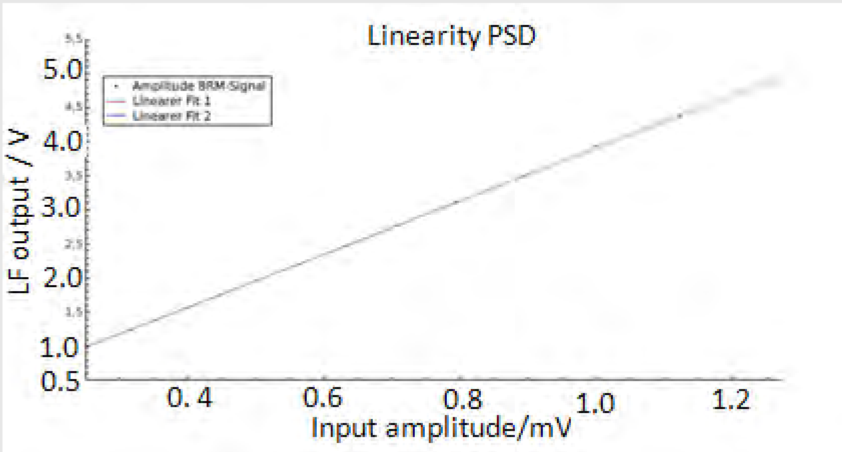
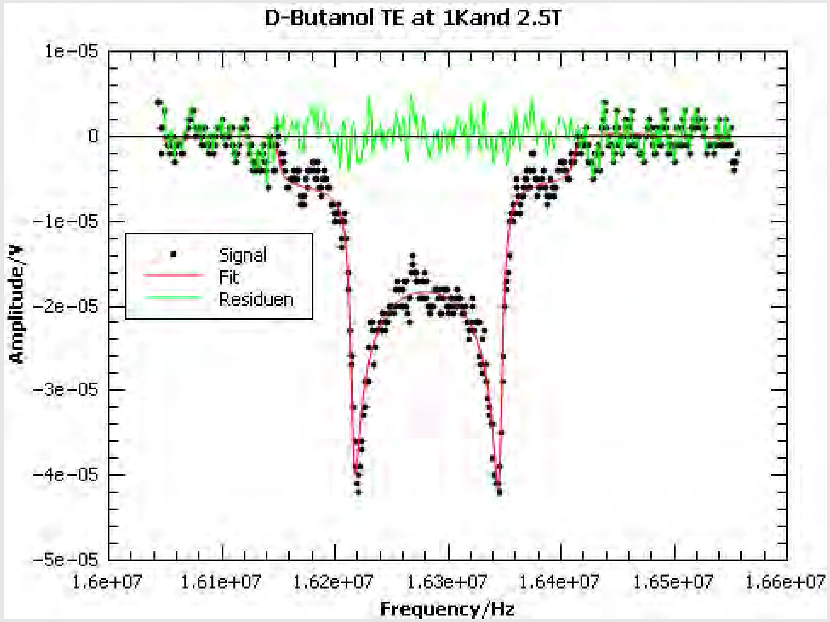
Bochum NMR box











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Summary / Outlook

- Bonn polarized target and internal polarization magnet development
- T_{1e} measurements of different radicals in different host materials and at various temperatures
- Polymer target preparation
  - Polyethylene / Polypropylene
  - C_8D_8 preparation as target material and performance
- Bochum NMR-Box

- T_{1e} for temperatures below 1K
- Further development of Polymer targets
- Bochum NMR box is going to be prepared for “series” production