High deuteron polarization in polymer target materials

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Content

1. Dynamic nuclear polarization (DNP)

2. The trityl radicals – progress for deuterated target materials

3. D-polymer materials: CD_2 and C_8D_8

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5. EPR investigation and Polarization results for
   - — radiation doped CD_2
   - — trityl radical doped C_8D_8

6. Conclusion and outlook

Polarized Solid Targets

- Polarized solid targets are used in particle physics experiments since more than 50 years.

- Concerning polarized solid targets, most important quantities as input:

$$P_t = \text{target polarization}$$

$$f = \textit{dilution factor} = \frac{\#\text{polarizable particles}}{\#\text{all particles}}$$

Asymmetry -----measurement with polarized targets

$$A = \frac{1}{P_t} \cdot \frac{1}{f} \cdot \frac{N\uparrow - N\downarrow}{N\uparrow + N\downarrow}$$

The Principle of Dynamic Nuclear Polarization

- **Thermal Equilibrium (TE)**

$$P = \frac{\langle I_Z \rangle}{I_Z^{\max}} = B_I \left(\frac{\mu B}{2kT} \right) \quad \propto \left(\frac{B}{T} \right)$$

B/T	P_p[%]	P_d[%]	P_e[%]
2.5T/1K	0.25	0.05	93
15T/10mK	91	30	100

- **Dynamic Nuclear Polarization (DNP)**
  - Transfer of polarization from paramagnetic electrons to the nuclei
  - Parameters of DNP: temperature; magnetic field; microwave power; electron relaxation time; the relation of EPR linewidth and nuclear Larmor frequency…
- **Doping with paramagnetic electrons:**

~ 10^3 nuclei feeded by 1 unpaired electron from:

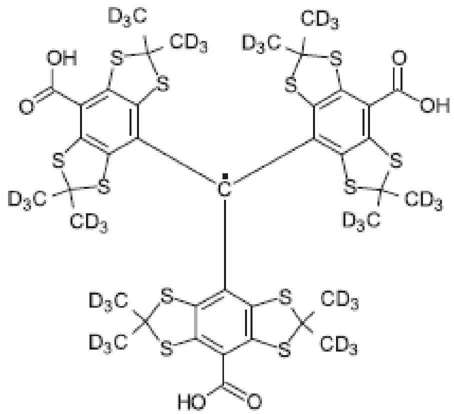
- Chemically stable radical → Solids
- Radiation induced defects → Solids

In the 1970 already 80-90% in protonated materials

Until 2003 40-50% in deuterated materials

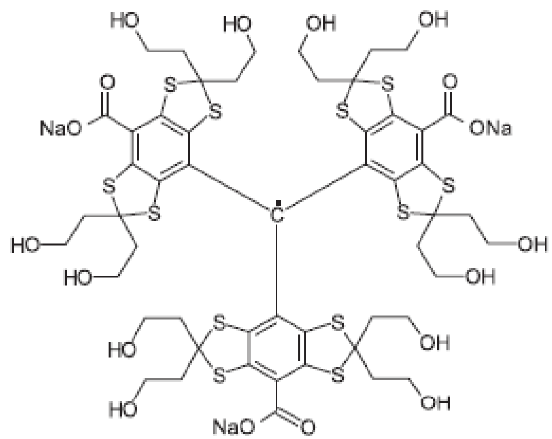
The Trityl Radicals (Malmö Group(Sweden); General Electric)

— Important Progress for Deuterated Materials



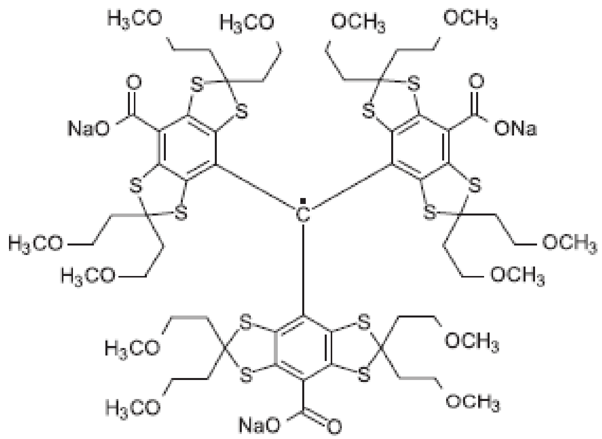
Finland D36'(AH110355 deutero acid form)used for butanol-d10

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

St.Goertz et al.NIMA 526 (2004)43

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

Important parameter: EPR linewidth

- Zeeman Energy of a free electron

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

- Contributions to the Electron Zeeman linewidth

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

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

Bochum measurements

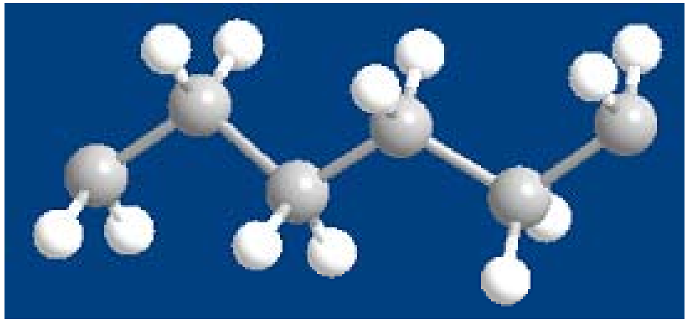
Material	Radical	$\Delta g/\bar{g}$ [10^{-3}]	FWHM [mT]	at 2.5T $P_{D,max}$ [%]
D-Butanol	EDBA	5.98 ± 0.03	12.30 ± 0.20	26
D-Butanol	TEMPO	3.61 ± 0.13	5.25 ± 0.15	34
D-Butanol	Porphyrexide	4.01 ± 0.15	5.20 ± 0.23	32
$^{14}ND_3$	$^{14}\dot{N}D_2$	$\approx 2 \ldots 3$	4.80 ± 0.20	44
$^{15}ND_3$	$^{15}\dot{N}D_2$	$\approx 2 \ldots 3$	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.

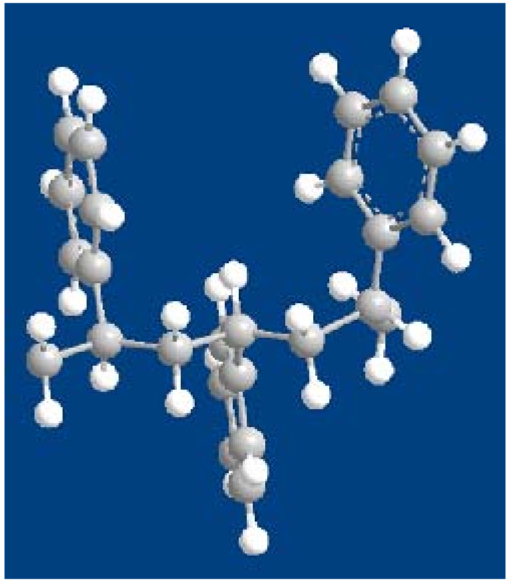
Result: The smaller the EPR linewidth, the higher the deuteron polarization value

Introduction to D-polymer materials

Poly(Ethylene-D4) CD_2

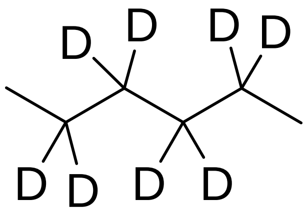


Styrene-D8, polymerized C_8D_8

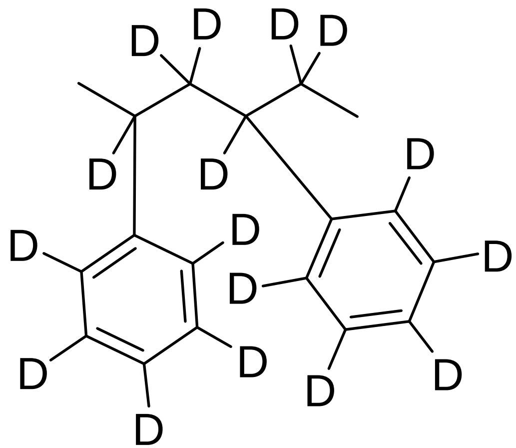


dilution factor

$$f = \frac{8 \text{ from D}}{24 \text{ from C} + 8 \text{ from D}} = 0.25$$



$$f = \frac{16 \text{ from D}}{96 \text{ from C} + 16 \text{ from D}} = 0.14$$



Motivation to use D-polymer materials

- **Spin physics**
  - Thin targets for scattering experiments at low energies
  - Polarized scintillator targets
- **Merits of CD_2, C_8D_8**
  1. High purity of D 0.98, 0.99
  2. D with spin 1 and C with spin 0
  3. Easy formable to any thickness at room temperature
- **Up to now the maximum polarizations of D-polymer**
  1. D-polyethylene **CD_2** : Paramagnetic Center---Irradiation

     35% at 6.5T/1K

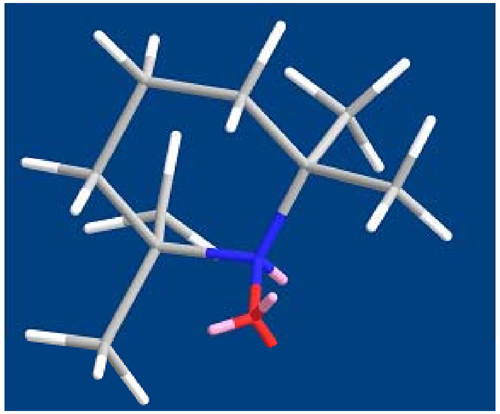
     D.G.Crabb, *Nucl. Instr. and Meth. A* 526, 56 (2004)
  2. D-Polystyrene **C_8D_8** : Paramagnetic Center---D-TEMPO

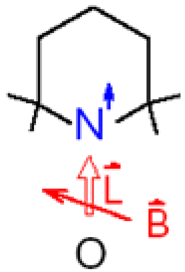
     40% at 2.5 T/100mK

     B.van den Brandt,et al., *Nucl. Instr. and Meth. A* 526, 53 (2004)

Doping methods for DNP

- **Mechanism of Dynamic Nuclear Polarization**
  Paramagnetic centers are needed
- **Chemical (Tempo, Trityl radical) doping**

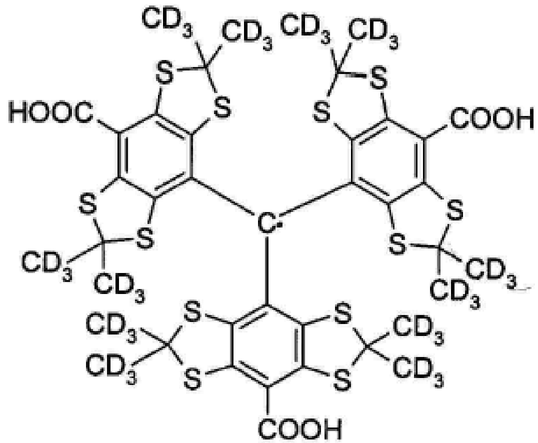




Melting point 36°C

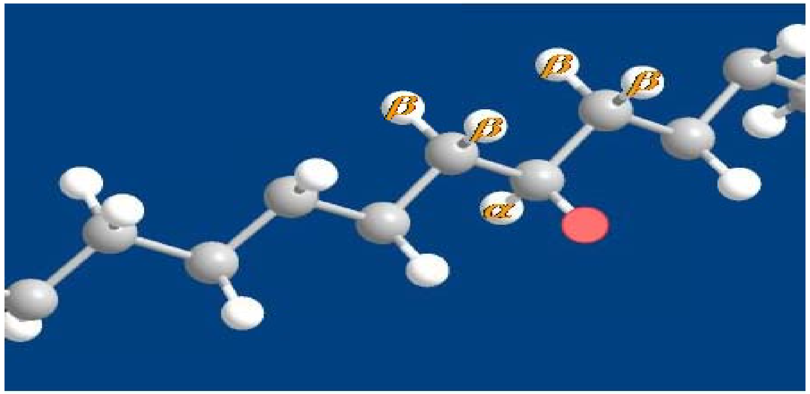
Boiling point 67°C

Tempo (stable free radical)

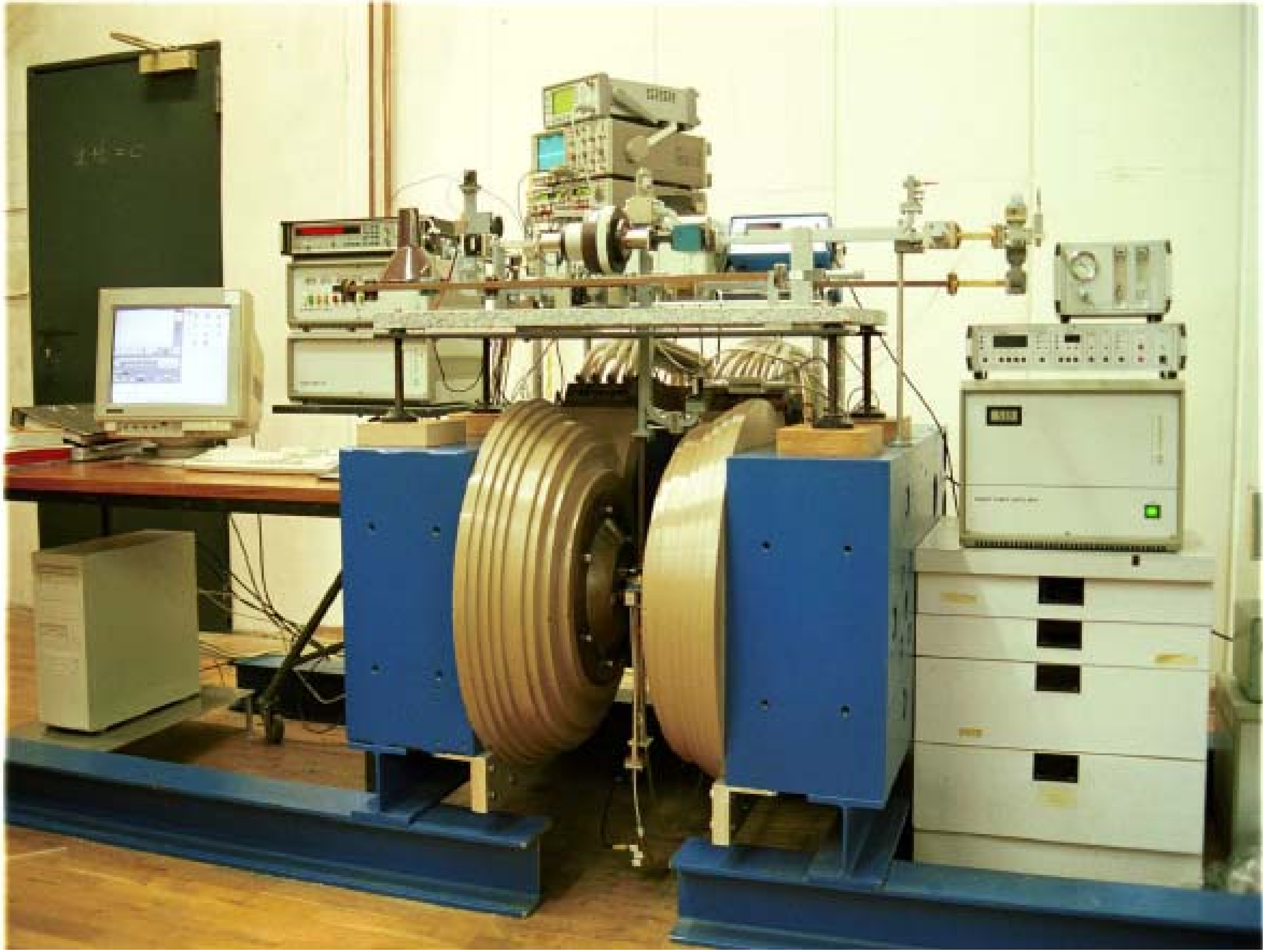


Trityl radicals Finland D36
(stable free radical)

- **Irradiation with electron beam**

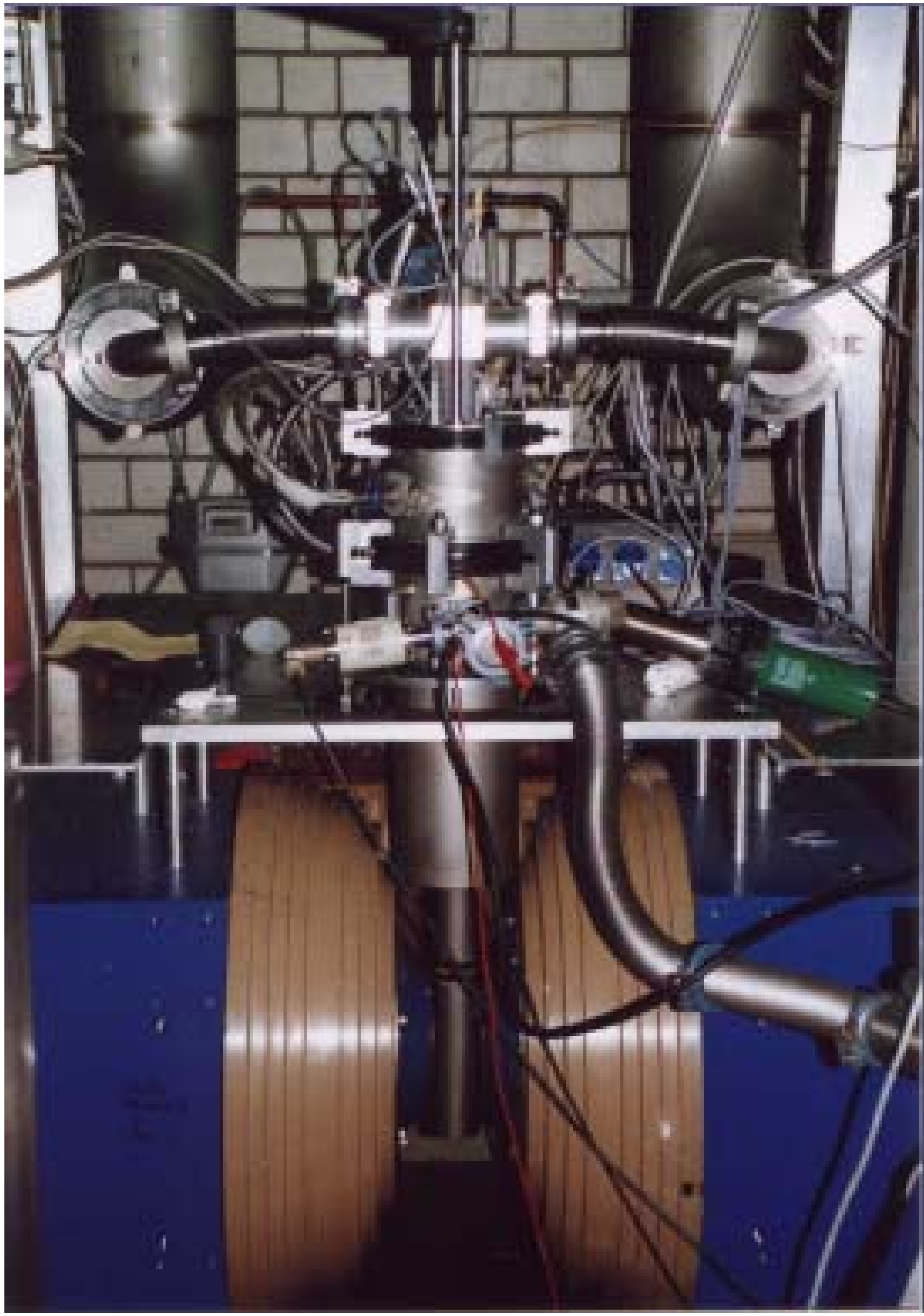


The Bochum EPR Apparatus



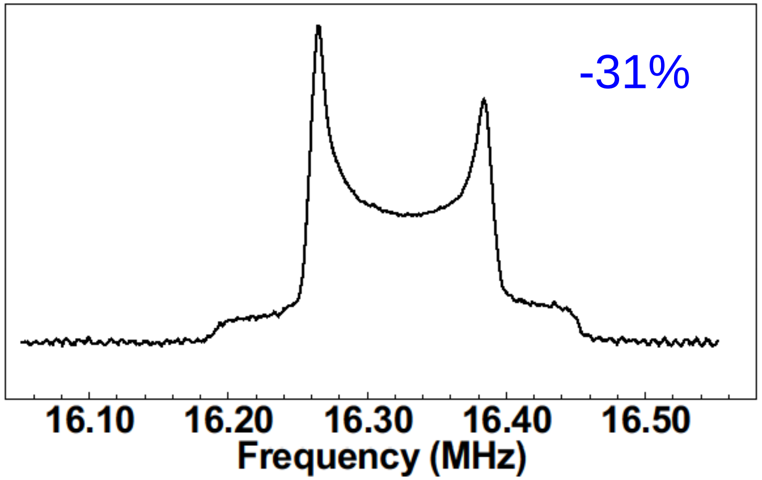
The Bochum DNP Apparatus

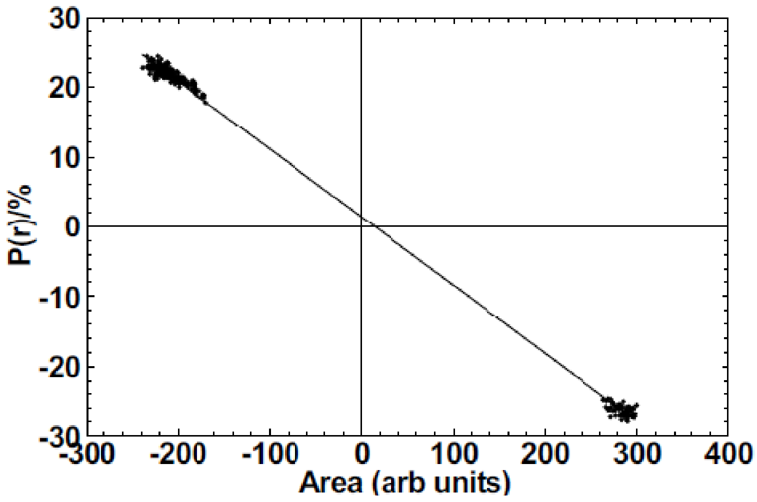
Magnet+cryostat

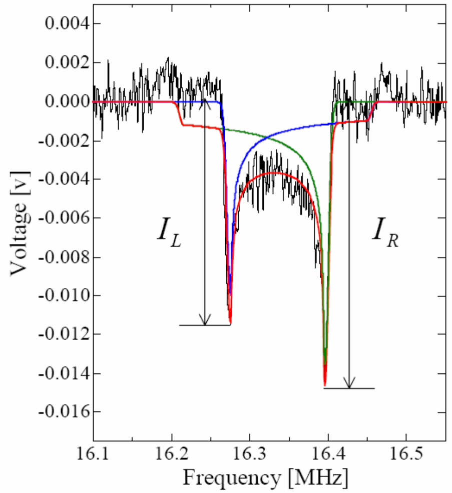


Polarization of radiation-doped CD_2

NMR Signal of Deuteron







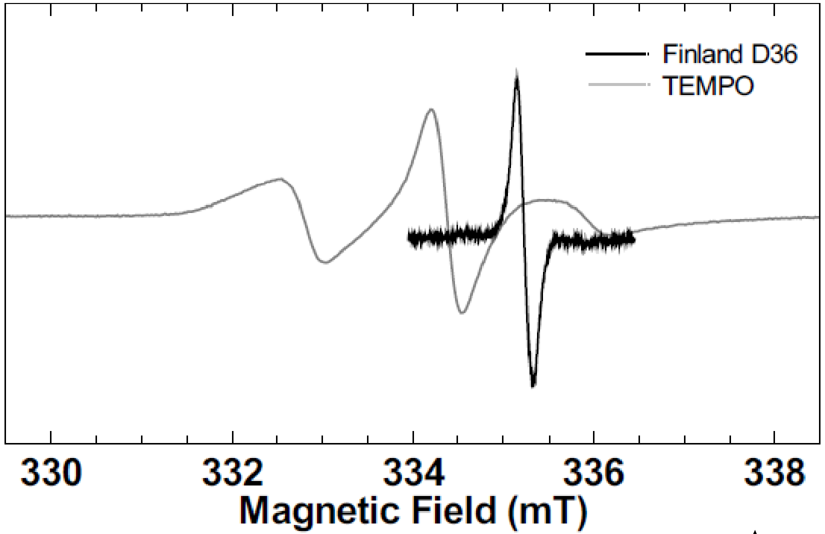
Temperature = 150mK *Magnetic field=2.5T*

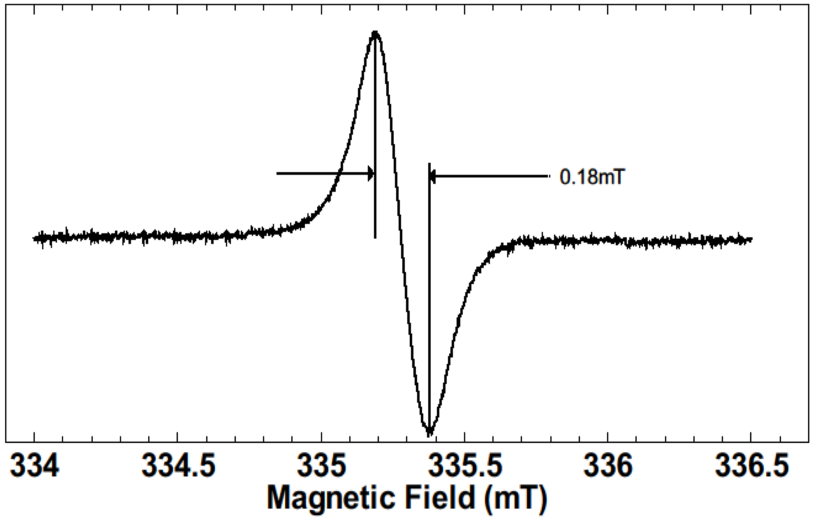
Dose [e^-/cm^2]	DNP Temp.[mK]	f_{mw}[GHz]	d-Pol[%]	$T_{build-up}$[min.]	f^+-f^- [MHz]
6.0×10^{15}	150	69.860 70.075	+21.0 -31.0	110	215

$$P = \frac{r^2 - 1}{r^2 + r + 1}, \quad r = \frac{I_R}{I_L}$$

* The large difference of positive and negative polarization values is still not understood.

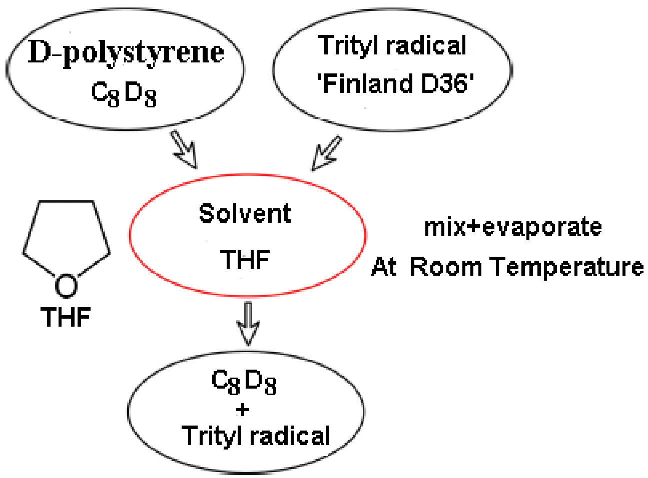
Preparation of trityl radical in C_8D_8





g-factor anisotropy: $\frac{\Delta g}{g} \sim 3.0 \times 10^{-4}$

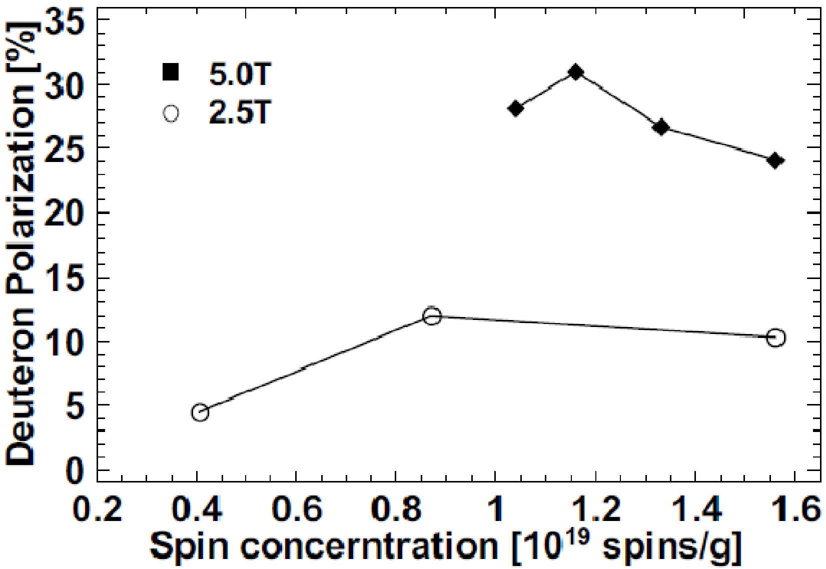
➢ Introduce Finland D36 Radicals in C_8D_8

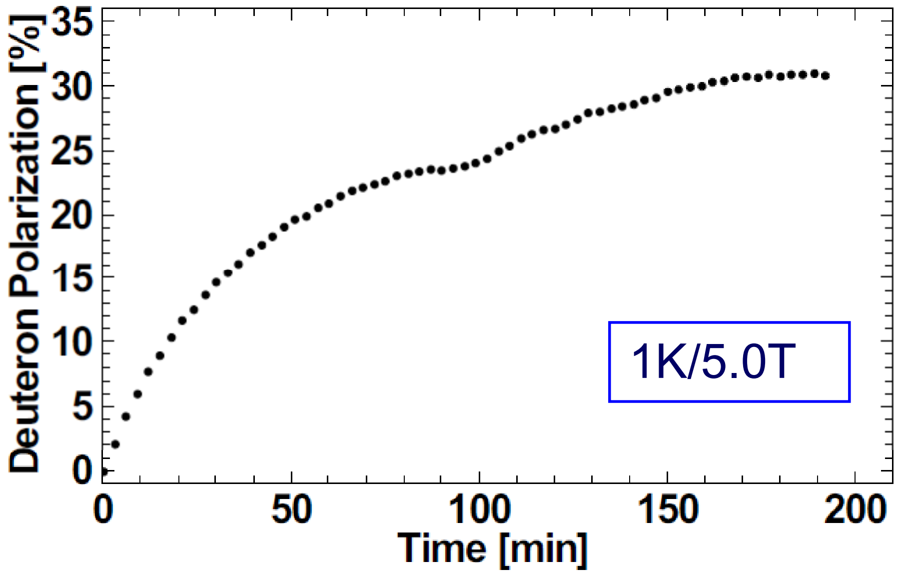


Homogenous and transparent

A thin foil (70μm)

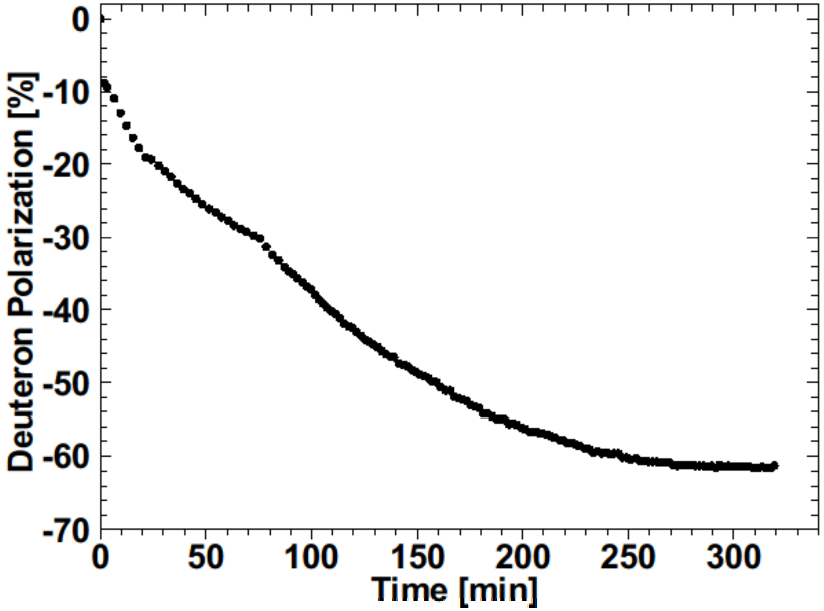
Polarization of Finland D36-doped C_8D_8

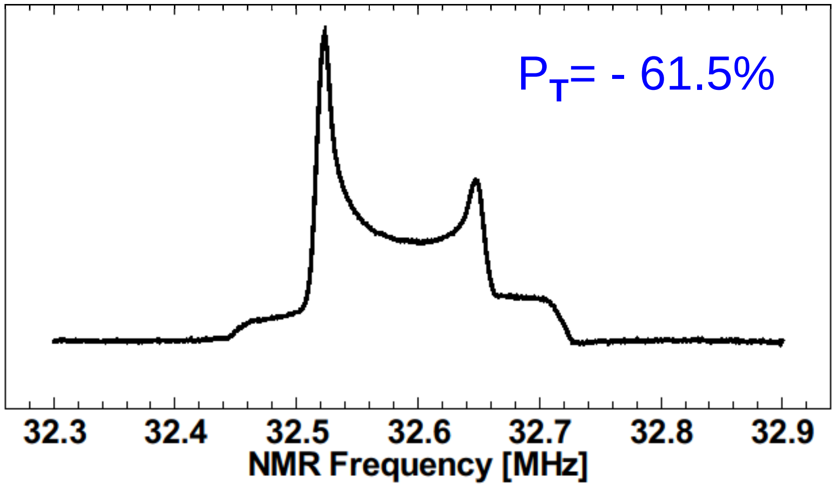




Spin conc. (spins/g)	Mag. Field (T)	$T_{build-up}$ (min)	$T_{1,d}$ (min)	Microwave Freq. (GHz)	d-pol. (%)	f^+-f^- (MHz)
0.87×10^{19}	2.5	76	80(T=1.01K)	69.877	+10.2	56
				69.933	-12.5	
1.16×10^{19}	5.0	47	139(T=0.99K)	139.736	+29.5	92
				139.828	-31.0	

Polarization of Finland D36-doped C_8D_8





Temperature = 400mK   Magnetic field= 5.0 T

Sample	MW (GHz)	d-pola. (%)	$T_{l,d}$ (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

Li Wang, et al., NIM A 729 (2013) 36

Deuteron Polymer Polarizations

Material	Doping	Magnetic field(T) 2.5 Polarization(%)	Magnetic field(T) 5 Polarization(%)	Temperature	$T_{l,d}$ (min)	FWHM-bolometric (mT)
CD_2	Irradiation (8.0×10^{15} e^-/cm^2)	+21.1 -31.1		150mK		
CD_2	Tempo (3.0×10^{19} spins/cm^3)	+11.1 -9.3		330mK		
C_8D_8	Tempo (2.3×10^{19} spins/g)	+7.3 -7.7		1 K	12	6.73 *(2.5T)*
C_8D_8	Trityl (1.16×10^{19} spins/g)	+11.8 -12.3		1 K	24	1.87 *(2.5T)*
C_8D_8	Trityl (1.16×10^{19} spins/g)		+29.5 -31.0	1 K	139	3.06 *(5.0T)*
C_8D_8	Trityl (1.16×10^{19} spins/g)		-61.5 +56.1	400mK	863	

Conclusion & Outlook

1. Irradiated D-polyethylene with a relative high dilution factor can be polarized to about 30%- 35% at 2.5T/150mK.
2. Chemically doped D-polystyrene with trityl radical can be polarized to more than 30% at 5.0T/1K and more than 60% at 5.0T/400mK with potential to values higher than 80%. But the dilution factor is much lower than that of D-polyethylene.
3. A approach for D-polyethylene with trityl radical doping is needed.

Thanks for your attention!