液体^{3}Heに対する能動核偏極

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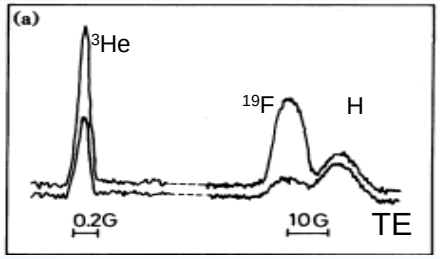
POLARIZING LIQ. ^{3}He in LITERATURES

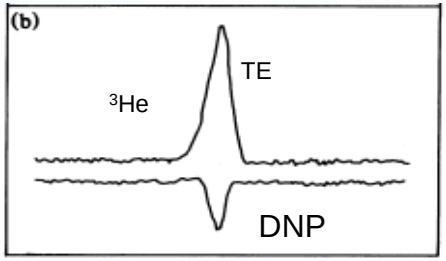
L.W.Engel and Keith DeConde, Phys. Rev. B **33**, 2035 (1986).

- **powdered sucrose charcoal** (with paramagnetic centers)
- Polarization transfer: **electron → ^{1}H → ^{3}He**
- Small enhancement obtained
  - +18% enhancement w.r.t. TE signal amplitude at T=1.8K,B=182G
- relaxation time T_1 measured to be 1.02sec

A.Schuhl, S.Maegawa, M.W.Meisel, M.Chapellier, Phys. Rev. Lett. **54**, 1952 (1985).

- **fluorocarbon beads** (paramagnetic centers produced in production process)
- Polarization transfer: **electron → ^{19}F→^{3}He**
- Polarization enhancements observed





T=250mK, B=300G

PSI group also performed the similar investigation. No enhancement was observed.

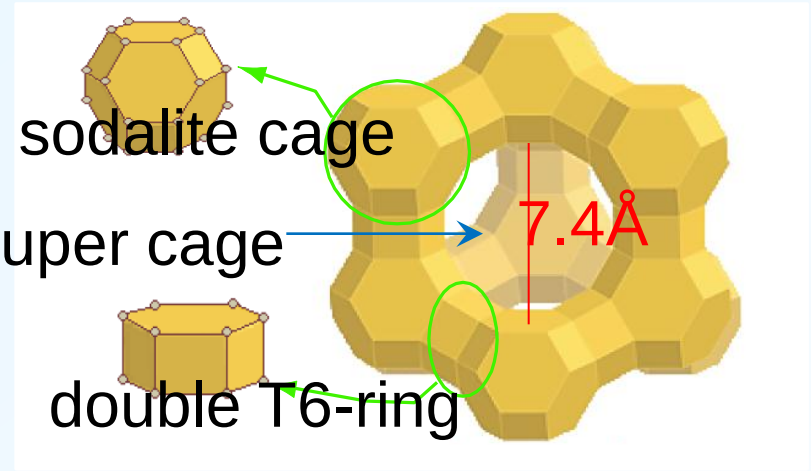
POLARIZING LIQUID ^{3}He by DNP

- Challenge in a different way
- **Direct spin coupling between electron and ^{3}He**
- Embedding a free radical into a porous material

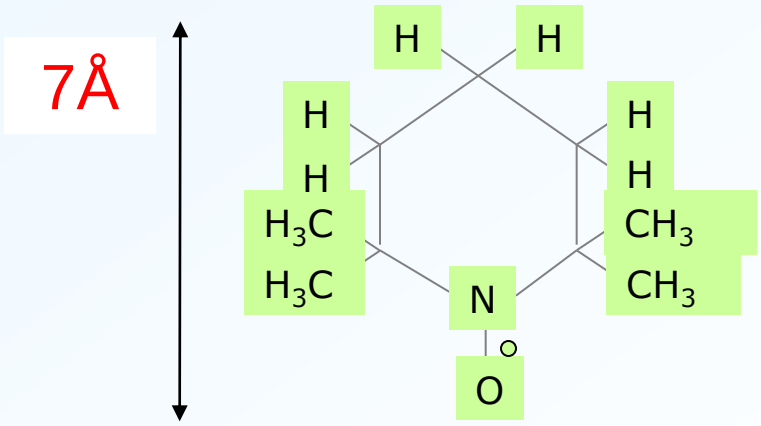
  (electron spin density controllable)
- Filled with liquid ^{3}He
- Proceed DNP with microwave
  - Porous material → **zeolite**
  - Free radical → **TEMPO**

ZEOLITE and TEMPO

Zeolite($Na_nAl_nSi_{(192-n)}O_{384}\cdot 240H_2O_{(n=48\sim76)}$)



- NaY type zeolite (n=51)
  - Super Cage
    - ✓ Max dia.: 13Å
    - ✓ Window dia.: 7.4Å
  - 4.7×10^{19} super cages/g
  - ^3He(dia.:3Å) → ≈80 ^3He can get in one super cage
- TEMPO(2,2,6,6-tetramenthyl-piperidinyl-1-oxyle)
  - Melting point: 36 ℃
  - Boiling point: 67 ℃
  - Molecule size: ~7Å



EMBEDDING TEMPO into Zeolite

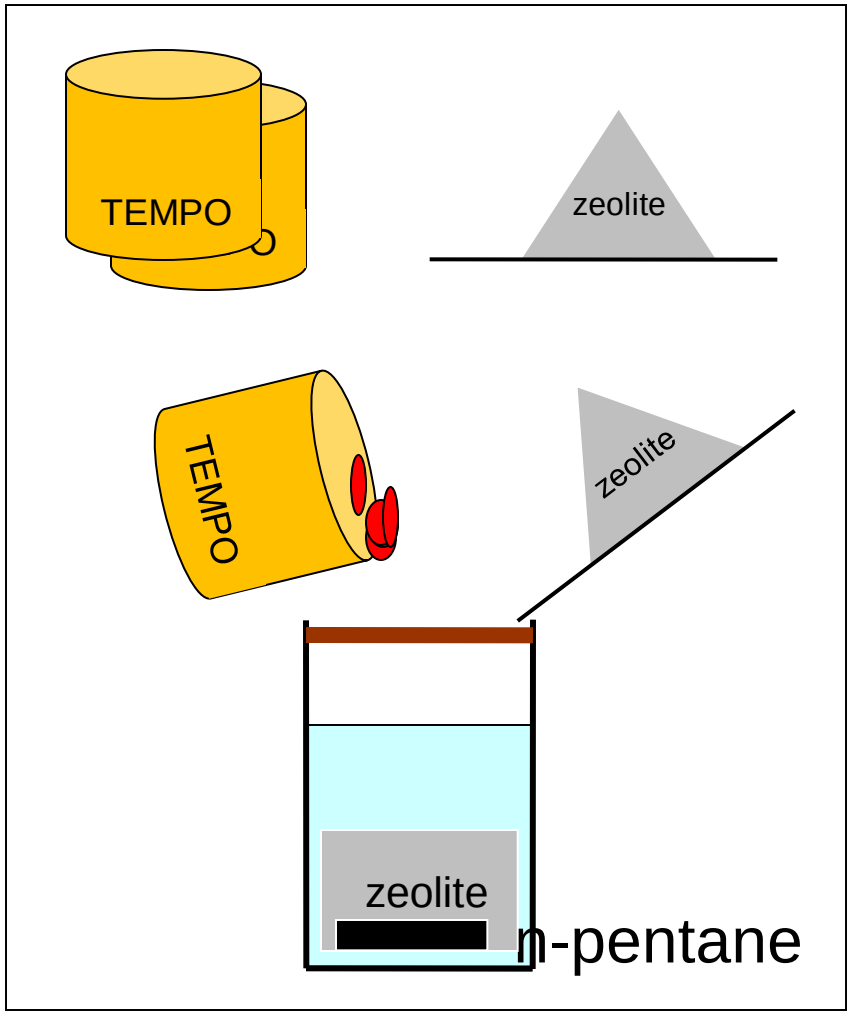
- ✓ Preparation : Desiccate zeolite at 500 ºC for 8 hours
1. Dissolve TEMPO in n-pentane
2. Add zeolite to n-pentane solution
3. Stir them for 8 hours in a sealed vessel
4. Evacuate n-pentane in a vacuum container

recipe

TEMPO:3~6mg

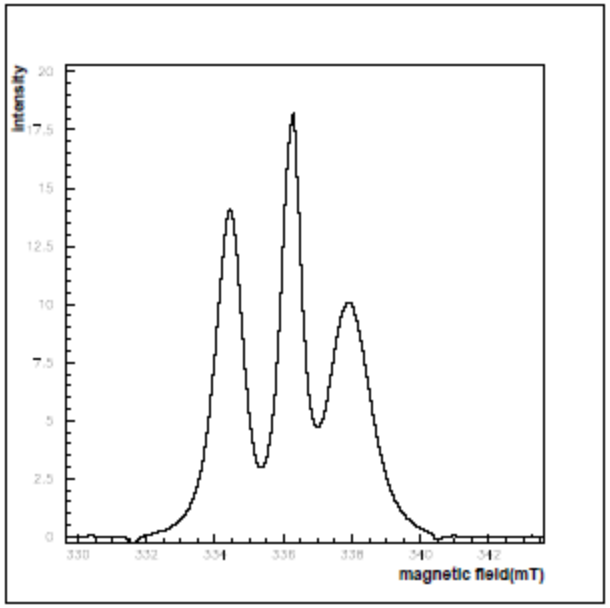
Zeolite:5~10g

n-pentane:50~100cc



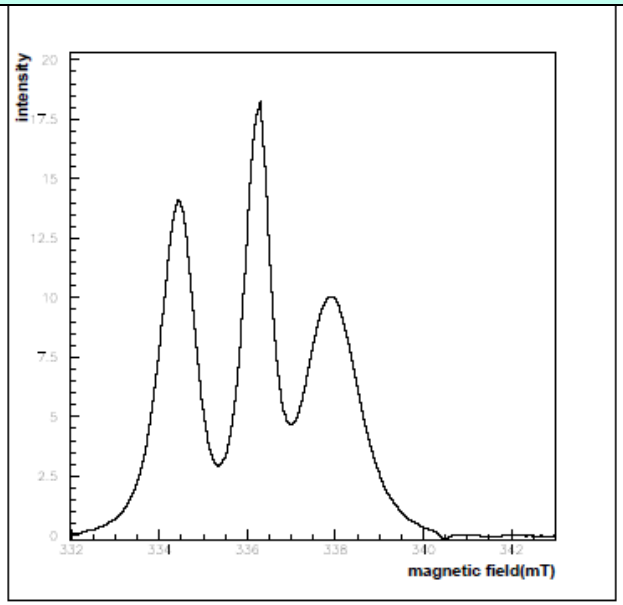
ESR OF TEMPO

TEMPO dispersed In zeolite



$4.5 \times 10^{18} \mathrm{spins/cc}$

TEMPO dissolved in n-pentane

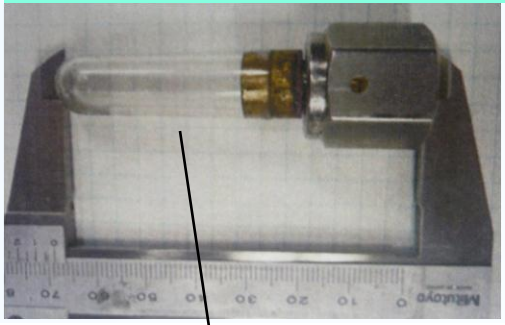


Similar ESR spectra

Uniform dispersion of TEMPO molecules in zeolite

SETUP OF THE TEST

Sample cell made of PET tube (2.5cm^3)



Dilution cryostat
(worked as a ^{4}He or ^{3}He cryostat)

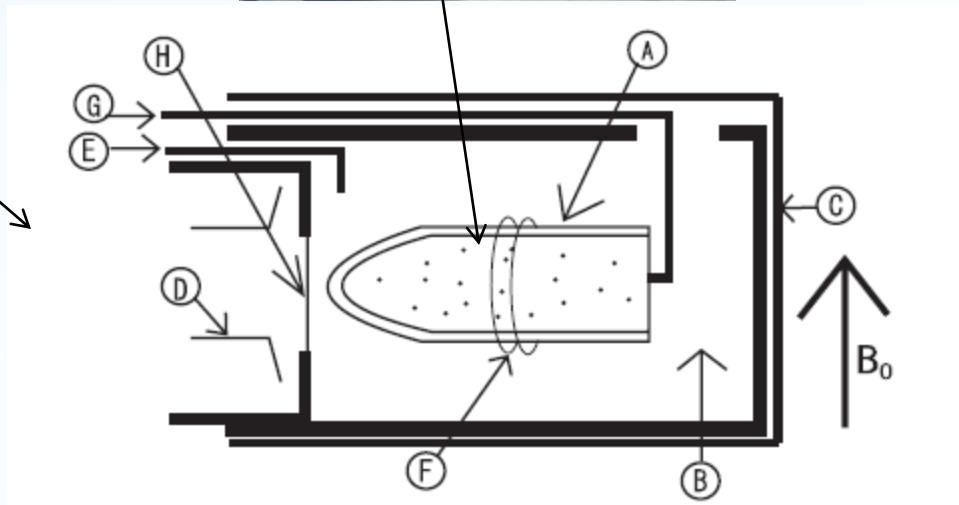
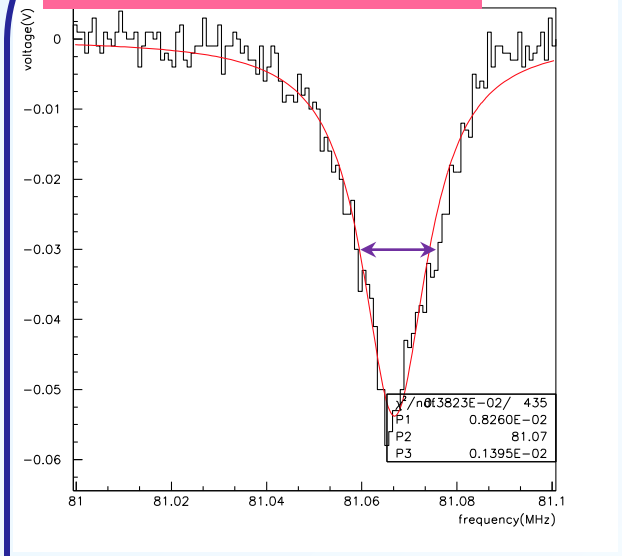


FIG. 1: Simplified diagram of the experimental setup showing A: the experimental cell; B: the ^{4}He bath made of teflon; C: the copper wall to form a microwave cavity; D: a microwave guide(R-band) providing a microwave through the mylar window(H); E: a liquid ^{4}He inlet;F: a 2-turn NMR coil, and G; a ^{3}He inlet.

^{3}He TE SIGNALs

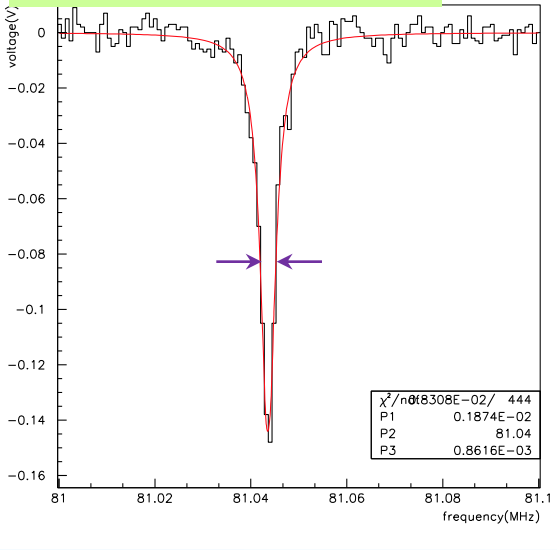
High concentration



spin density :1.3×10^{19}spins/cc
T=1.47K, B≒2.5T

FWHM=16.4 kHz

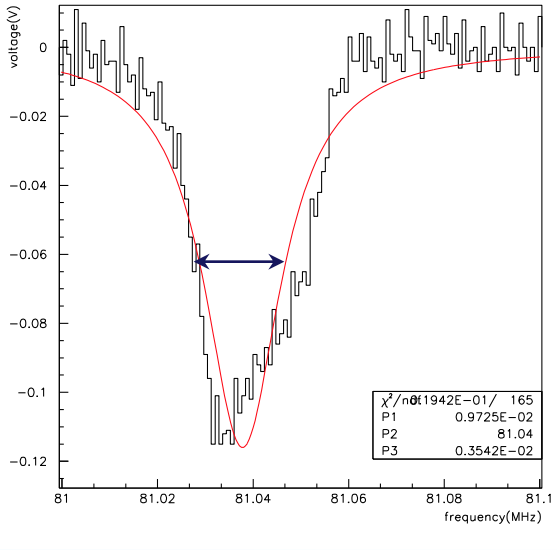
Low concentration



spin density :4.5×10^{18}spins/cc
T=1.54K, B≒2.5T

FWHM=3.6 kHz

Bulk 3He(without zeolite)



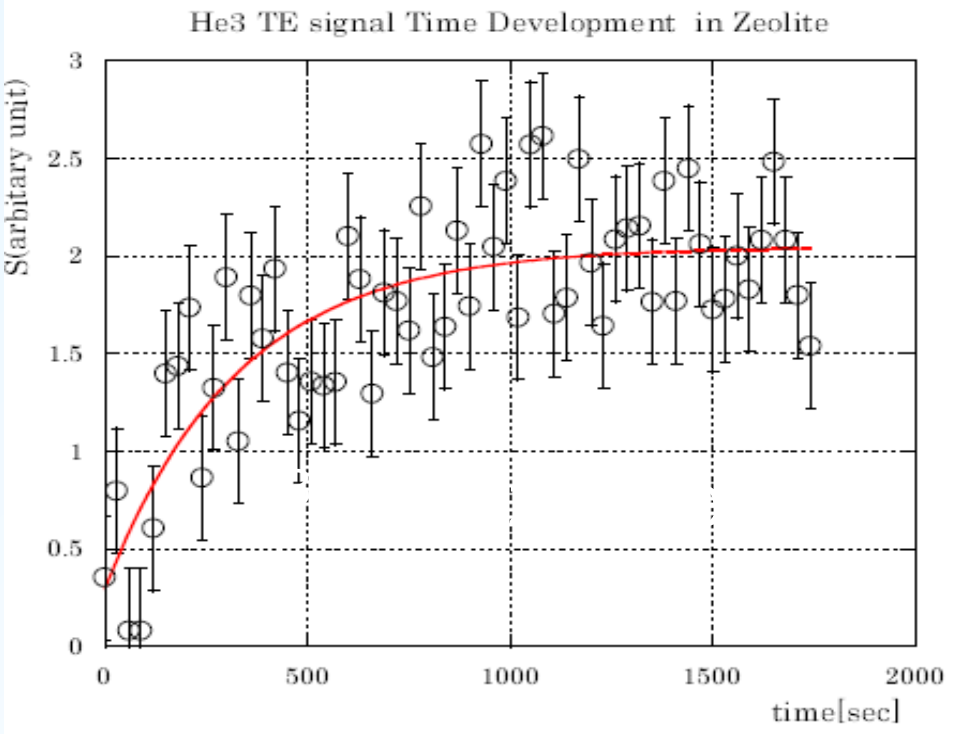
TE signal of liquid ^{3}He
T=1.42K ,B≒2.5T

FWHM=16.4 kHz

Signal fitted by Lorentzian

RELAXATION TIME

Development of ^{3}He TE signal
just after ramping up the magnet to 2.5T



High concentration

T=1.44K, B=2.5T, spin density: 1.3×10^{19} spin/cc

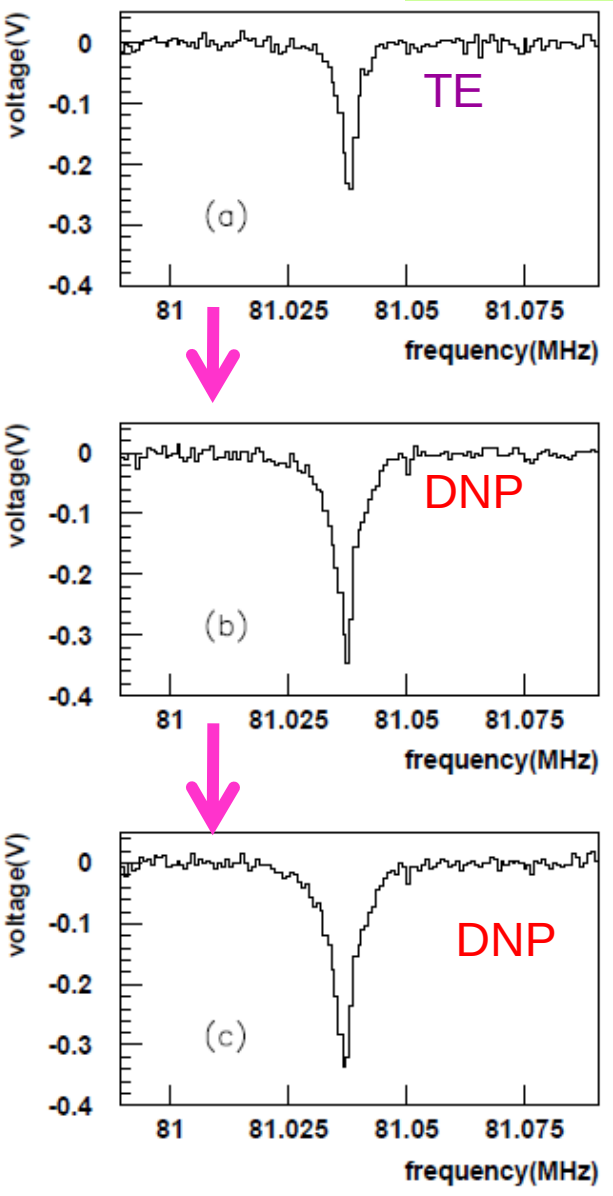
relaxation time: T_1=330sec

POL. ENHANCEMENT in DNP

4.5×10^{18}spins/cc.

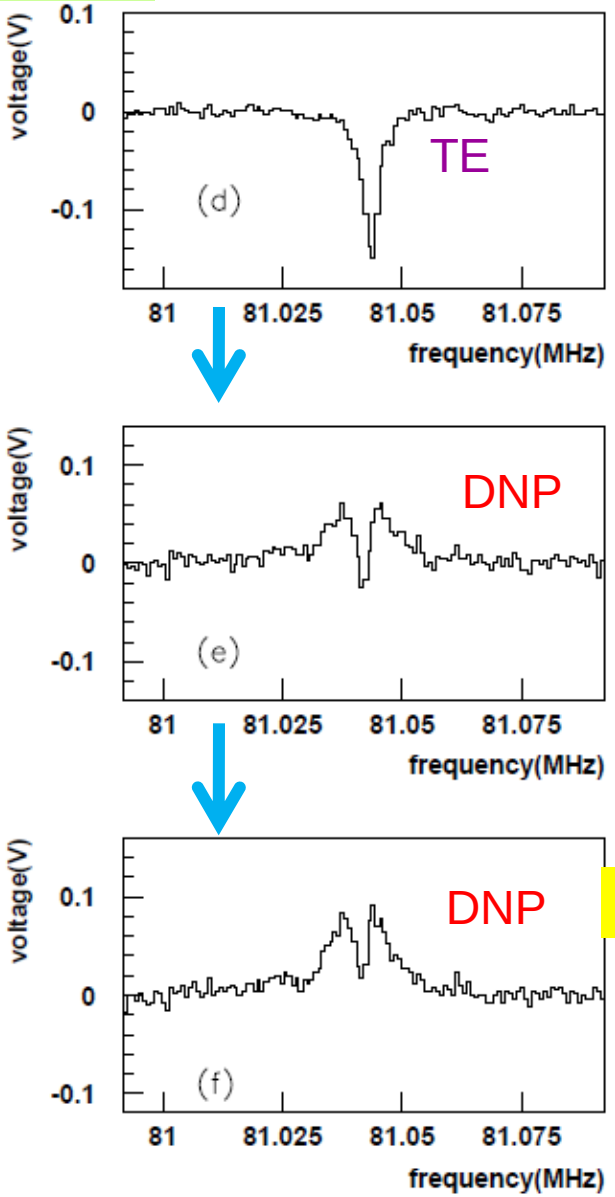
Low concentration

T=1.48K,
B≒2.5T



S/S$_{TE}$ =2.34

P=0.3%



T=1.53K,
B≒2.50T

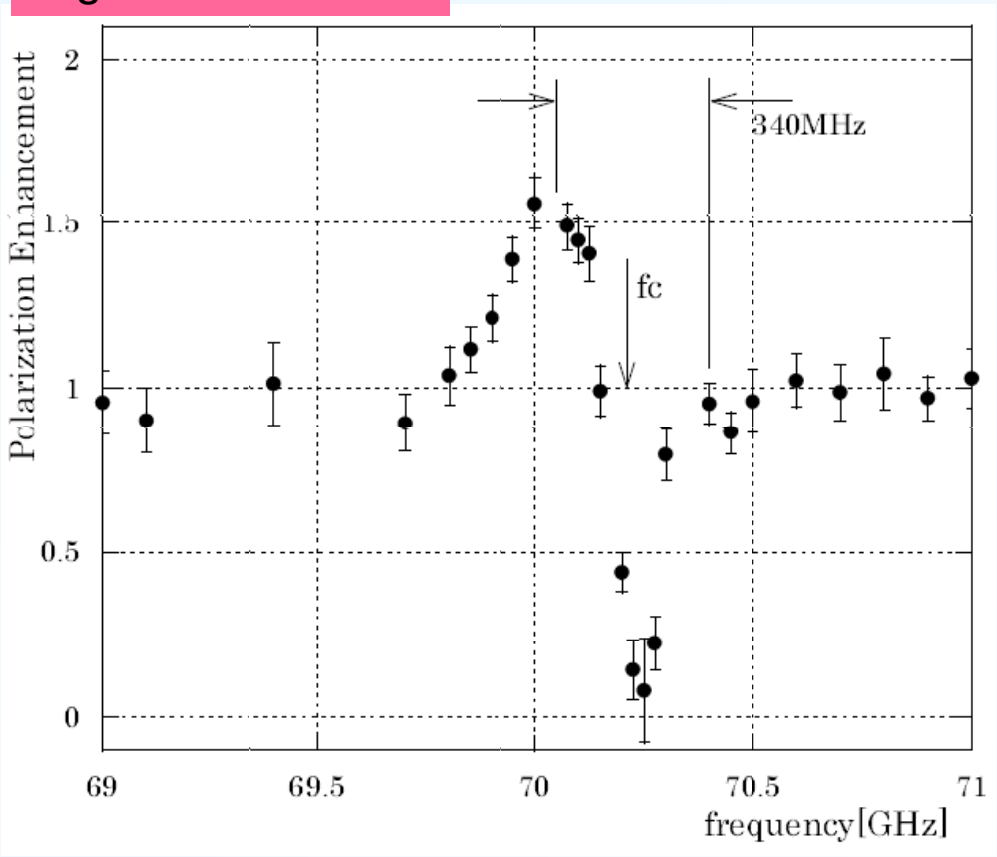
S/S$_{TE}$ =-1.59

P=-0.21%

Oct .15. 2011

MW FREQ. DEPENDENCE

High concentration



fc: ESR center frequency of TEMPO(=70.22GHz)

expected ESR line width for TEMPO is 340MHz at 2.5T

A little strange behavior
The line width narrower than expected

spin density:1.3×10^{19}, B≒2.5T

The results mentioned so far have been reported in PST2008

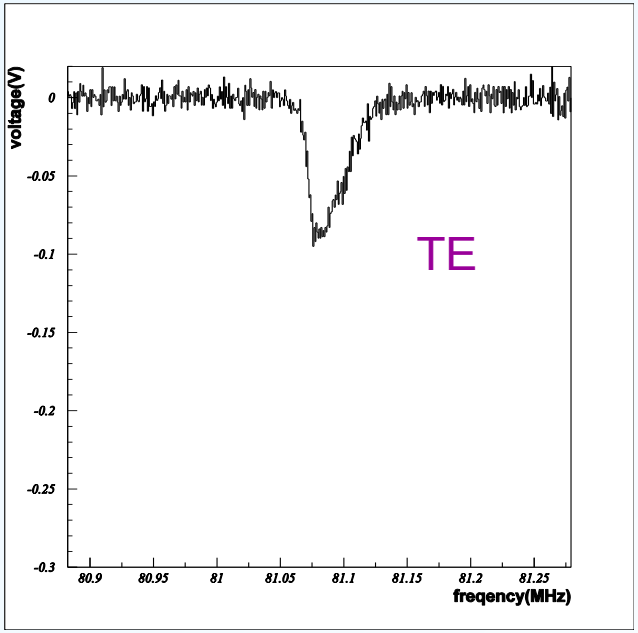
8 MONTHS LATER ....

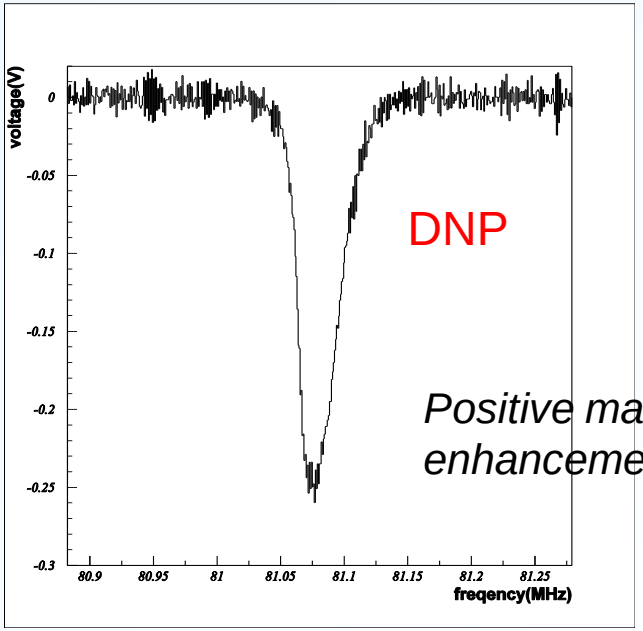
The samples had been kept in vacuum for 8 months at RT.

spin density: 4.5×10^{18} spins/cc (at previous test)

DNP test performed with the same sample

Low concentration





T=0.90K, B=2.5T,

T=1.1K,B=2.5T

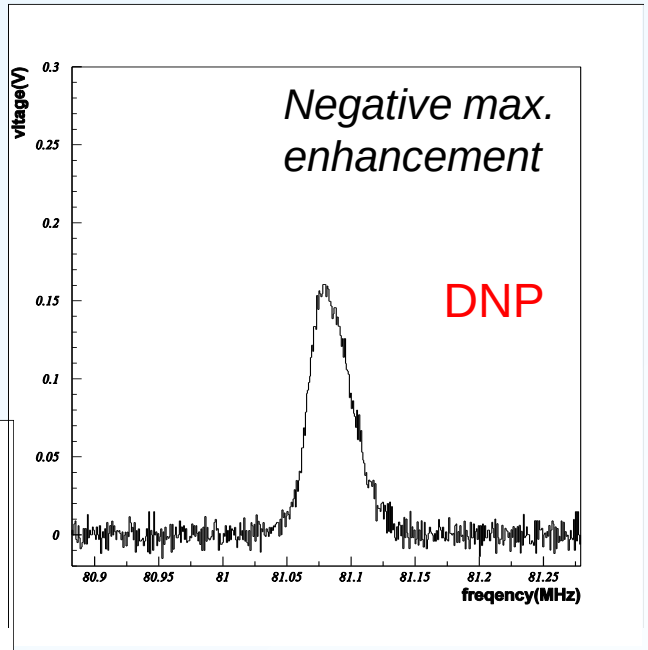
The test was done at lower temperatures around 1K

S/S_{TE} =3.09 ➔ polarization: P_+=0.67%

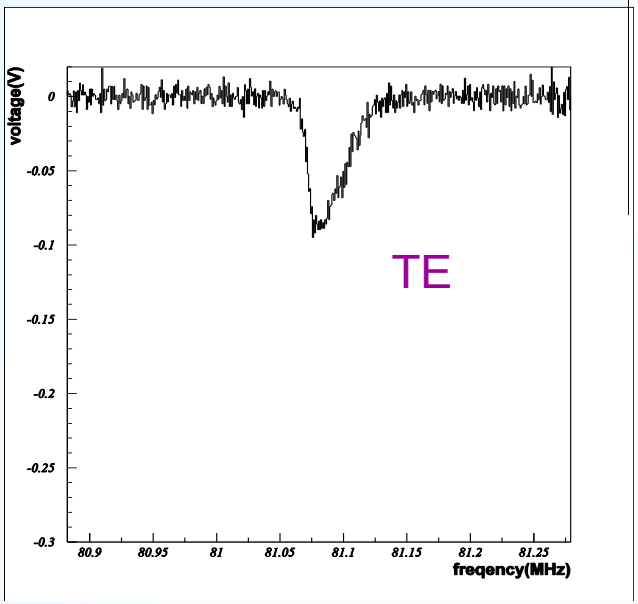
（ previous value: P_+=0.3%）

8 MONTHS LATER ...

Low concentration



T=1.0K,B=2.5T



T=0.90K, B=2.5T,

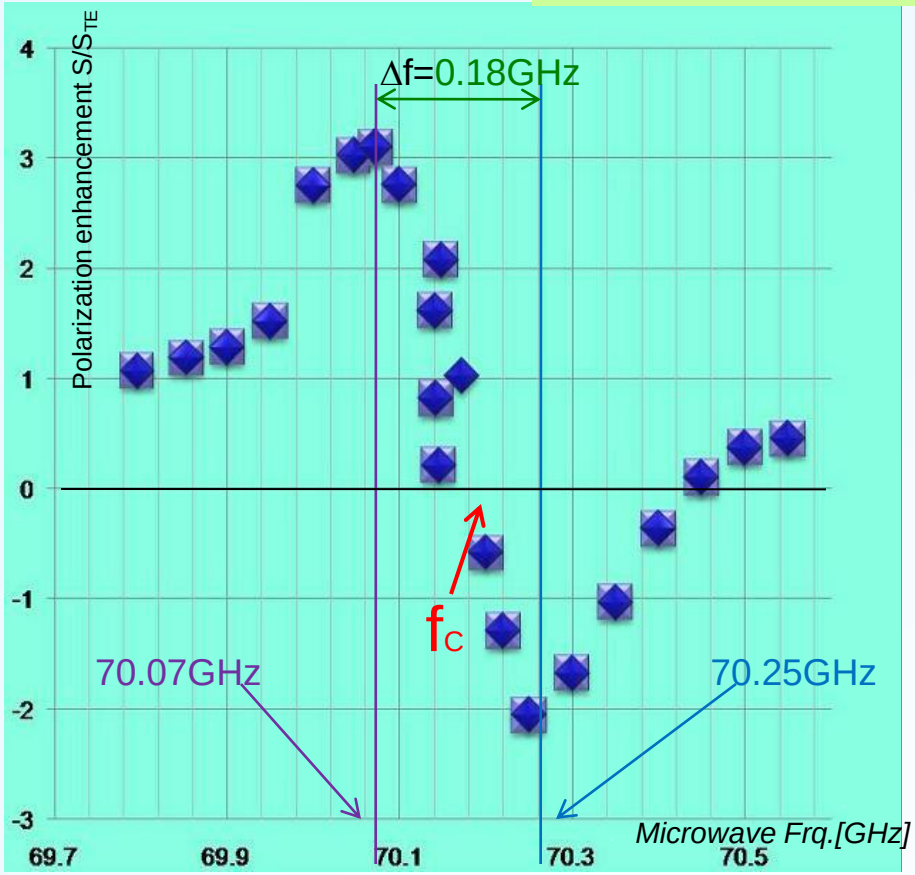
S/S_{TE} =-2.06

polarization: P_=-0.45%

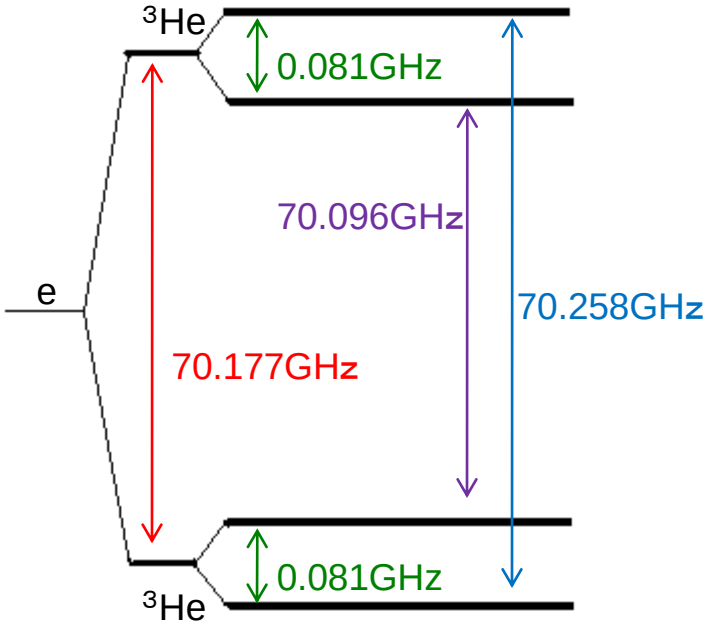
（prev. value: P-=-0.21%）

MW FREQ. DEPENDENCE

Low concentration



Energy level of electron&3He system at 2.5Tesla



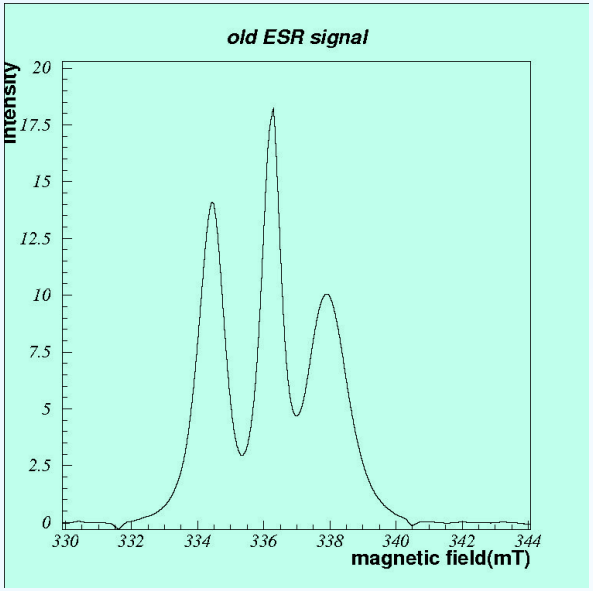
Δf=0.162 GHz for Solid State Effect

fc:ESR center frequency of TEMPO (=70.177GHz)

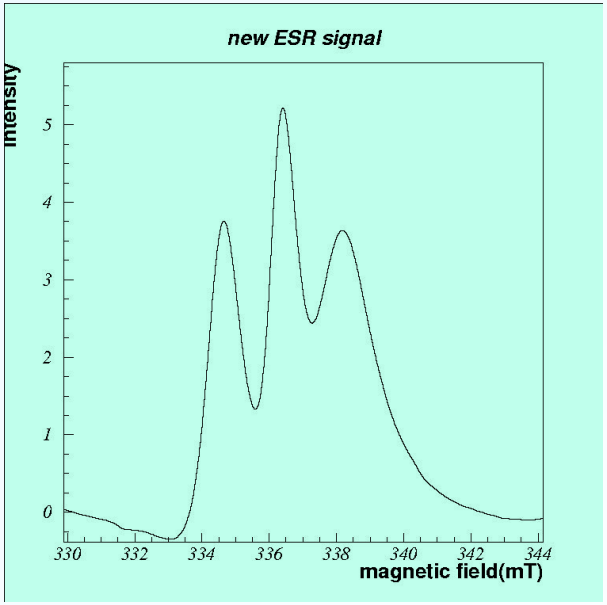
"Solid state effect" appeared with "liquid ^{3}He" !

CHANGE in ESR SPECTRUM

Feb.23.2007



Nov.28.2007(8 months later)

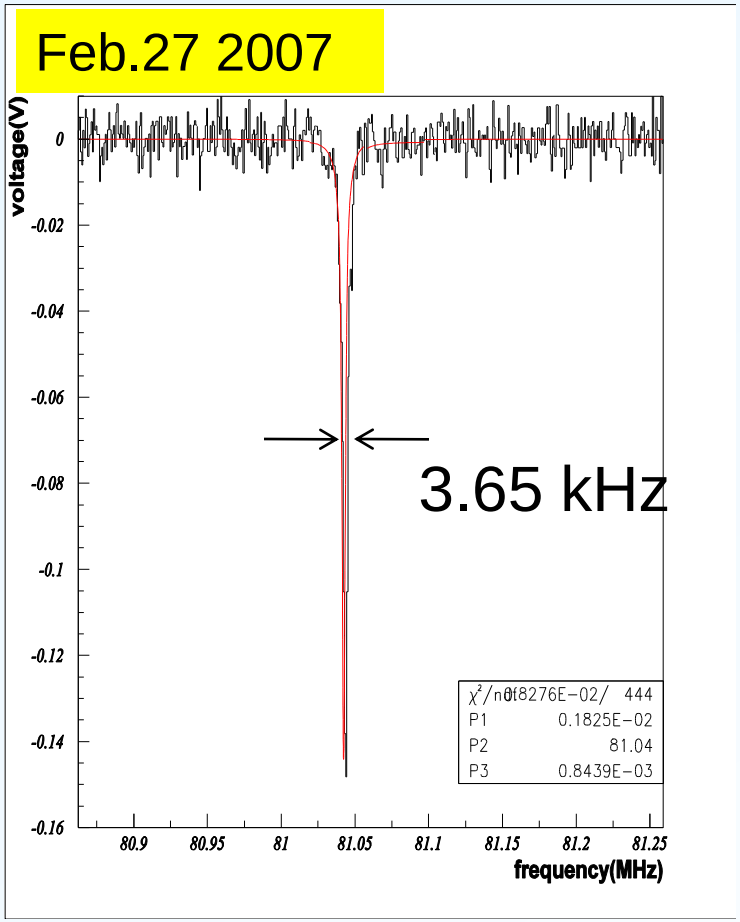


Spin density: 4.5×10^{18} spins/cc 0.93×10^{18} spins/cc

- spin density decreased : 1/5 w.r.t. previous value
  ➔ reasonable for storage in vacuum
- However, broader ESR observed
  ➔ localization(gathering) of TEMPO ?

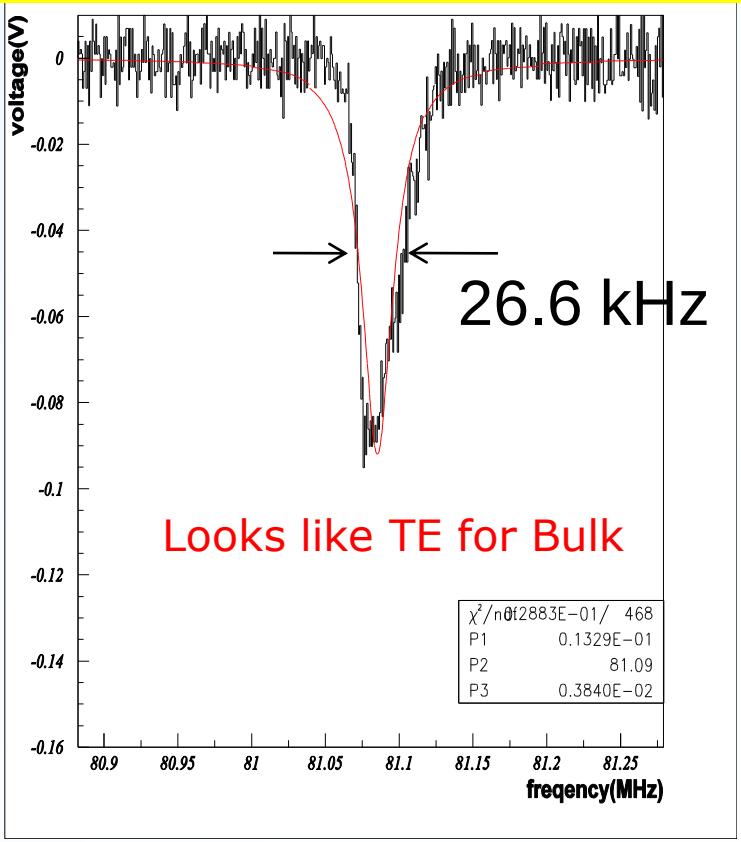
CHANGE in ^{3}He TE SIGNAL

Feb.27 2007



4.5×10^{18} spins/cc, T=1.5K, B=2.5T

Nov.09.2007 (8 months later)



0.93×10^{18} spins/cc, T=1.1K,B=2.5T

What happened?

Less concentration of TEMPO → more space for ^{3}He

→ spin-spin interaction among ^{3}He ? or .....?

SUMMARY for LIQ. ^{3}He DNP

- Liq. ^{3}He was polarized in DNP with direct spin coupling between electron and ^{3}He
- However, the obtained polarization is very small.
- Solid state effect has been observed for liquid ^{3}He
- We still have a lot of things to be understood.
- Further systematic investigations are required.