DNP for polarizing liquid ^3He

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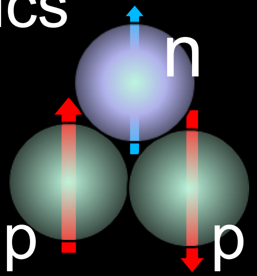
Background of the study

- Polarized ^{3}He targets have been used in various scattering experiments
  - > in ^{3}He only neutron is polarized
  - > a good target for the study of neutron characteristics
  - > studied only in gas targets



- Advantages of polarizing liquid ^{3}He
  - >Its fluidity may allow to make a polarized target with circulating polarized liquid ^{3}He
  - >Density gas(1atom):liquid=1:662
  - >Could be applied in many other fields (e.g. medical use, material science, chemistry, etc.)

How to polarize ^{3}He in liquid form

1. Brute force method

   >Polarized liquid is obtained by quickly melting polarized solid

   >55% polarization obtained in solid at 6.6T, 6mK, and 30bar, G.Bonfait et al. Phys.Rev.Lett. 53(1984)1092

   >However, it's difficult to make ^{3}He solid

5. Dynamic Nuclear Polarization (DNP)

   >spin-spin coupling between electron and nucleus

   >Transferring polarization of electrons to neighboring nuclei

   >able to obtain both positive and negative polarizations

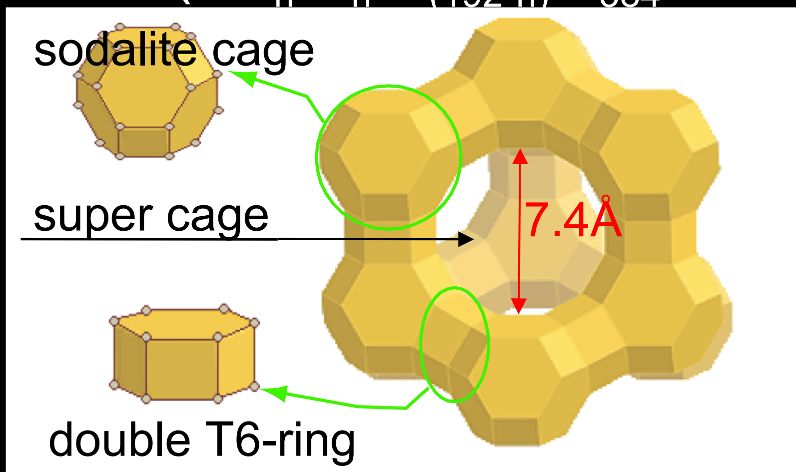
New method for polarizing liquid ^{3}He in DNP

- Direct coupling of a unpaired electron and ^{3}He
- Using unpaired electrons in a free radical
- Embedding the free radical into a porous material
- Filling the porous material with liquid ^{3}He
- Irradiating a microwave

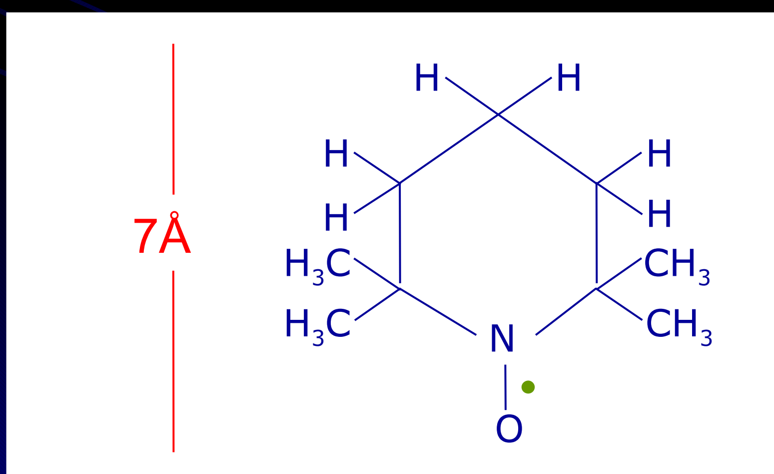
freeradical : TEMPO
porous material : zeolite

Zeolite and TEMPO

Zeolite($Na_nAl_nSi_{(192-n)}O_{384}\cdot 240H_2O$(n=48~76))



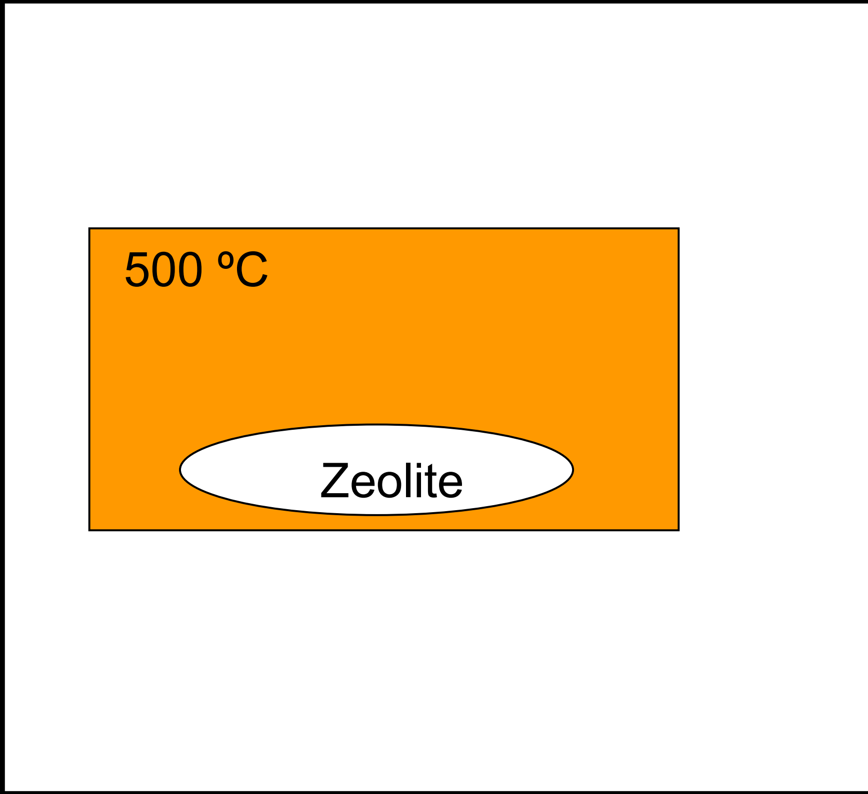
TEMPO



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

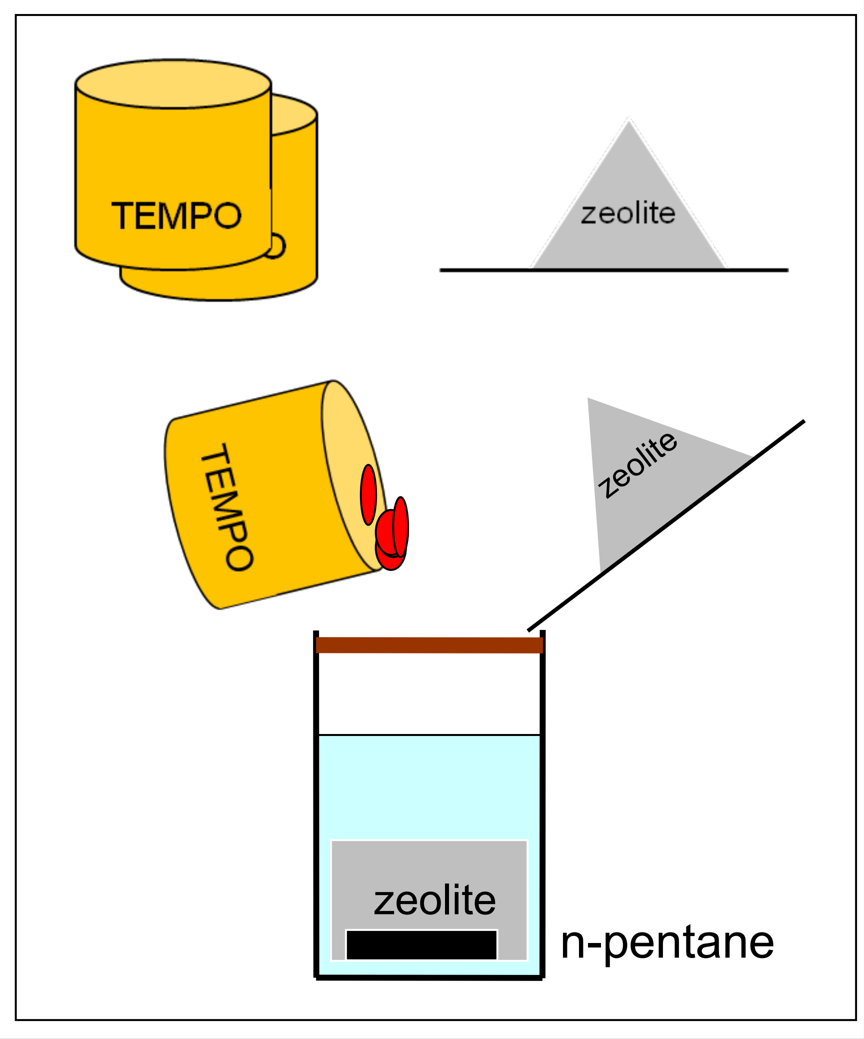
Embedding TEMPO to Zeolite

- ✓ 準備 Preparation : Desiccate zeolite at 500 ºC for 8 hours



Embedding TEMPO to Zeolite

- Dissolve TEMPO in n-pentane
- Add zeolite to n-pentane solution
- Stir n-pentane solution for 8 hours in a sealed vessel



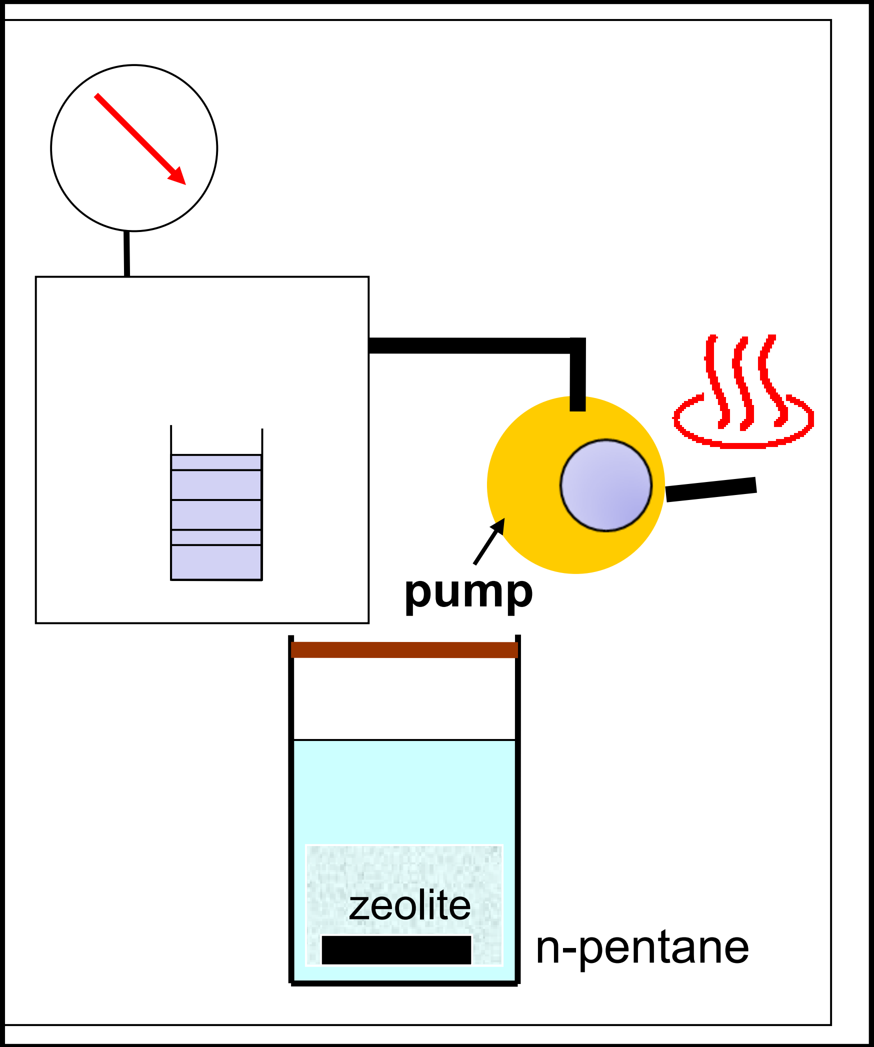
Embedding TEMPO to Zeolite

- Dissolve TEMPO in n-pentane
- Add zeolite to n-pentane solution
- Stir n-pentane solution for 8 hours in a sealed vessel
- Evaporate n-pentane in a vacuum container

TEMPO:3~6mg

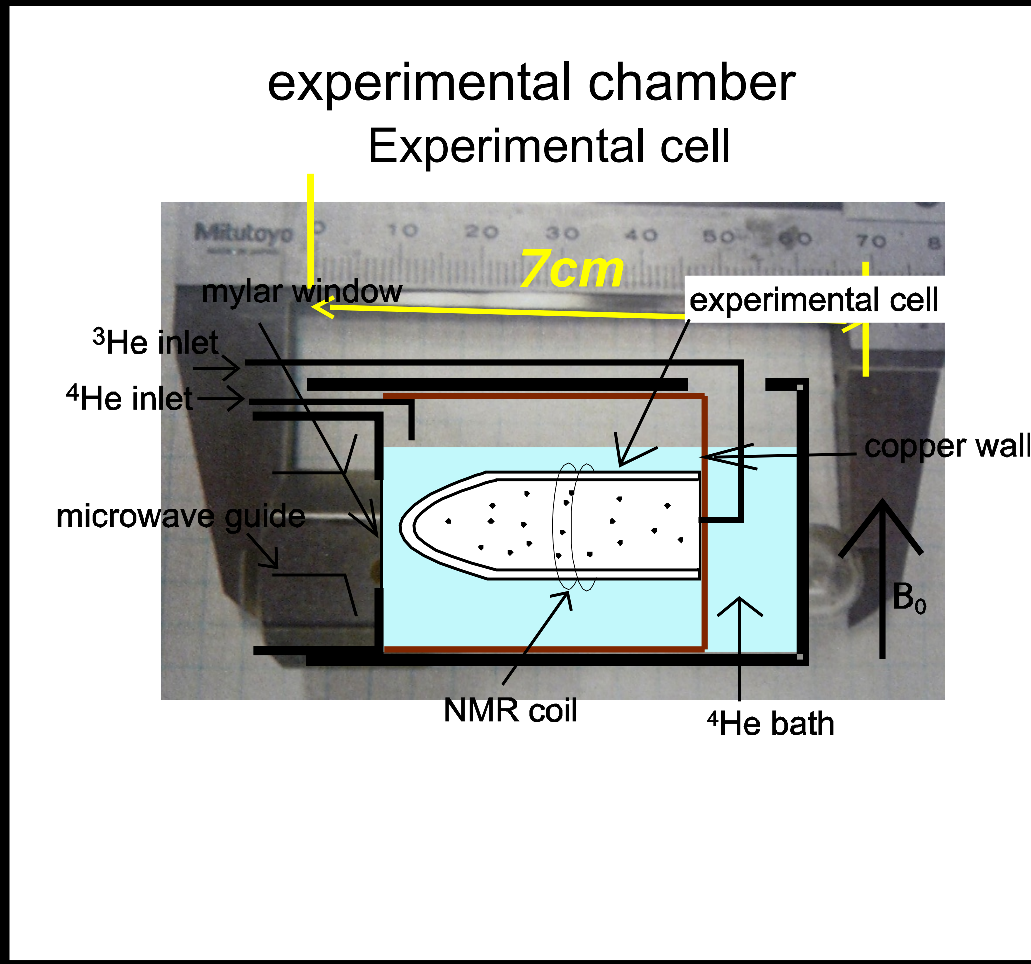
Zeolite:5~10g

n-pentane:50~100cc



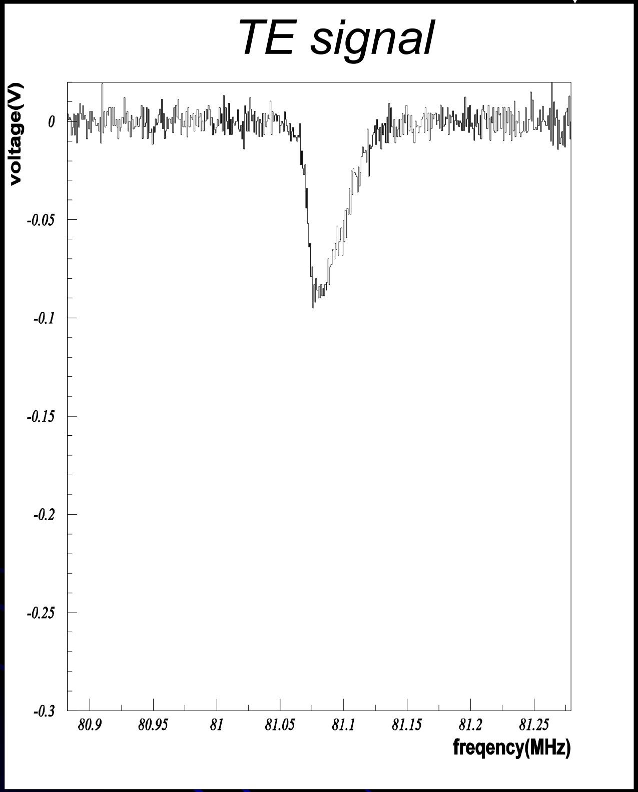
Experimental setup

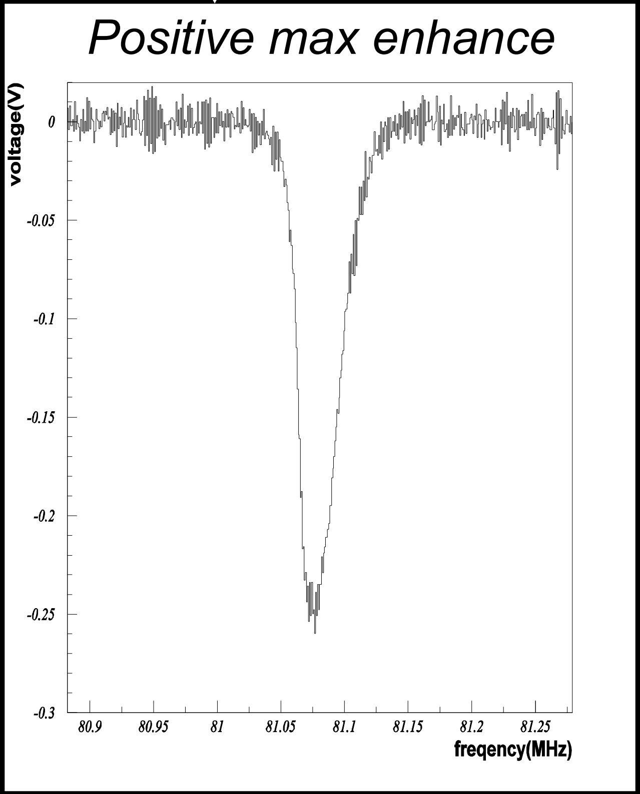
- Experimental cell :
  PET tube
  +
  VCR gas connector
- Volume : 2.5cc
  (L=35mm,
- Experimental cell filled with zeolite tightly and quickly



Positive enhancement by DNP

Liquid ^{3}He NMR signal





T=0.90K, B=2.5T,

Spin density: 0.93×10^{18} [spins/cc]

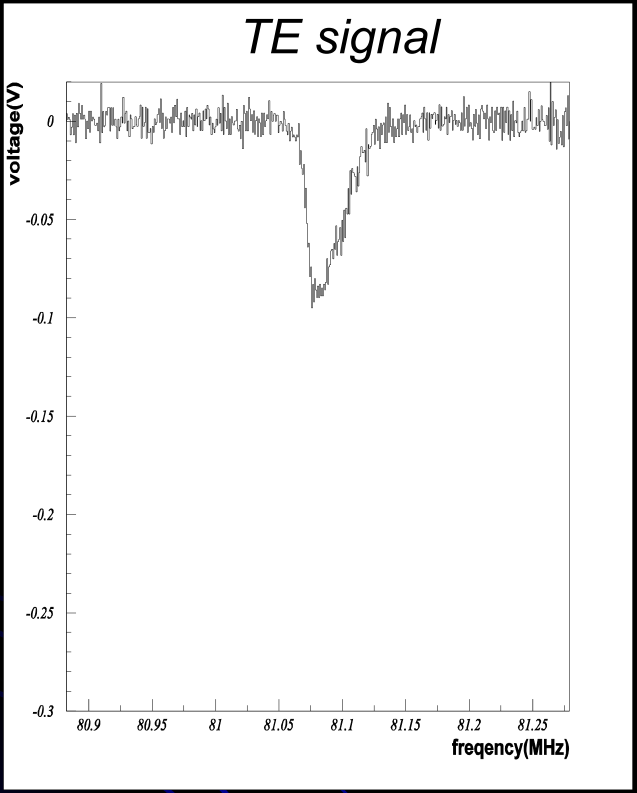
polarization 0.22

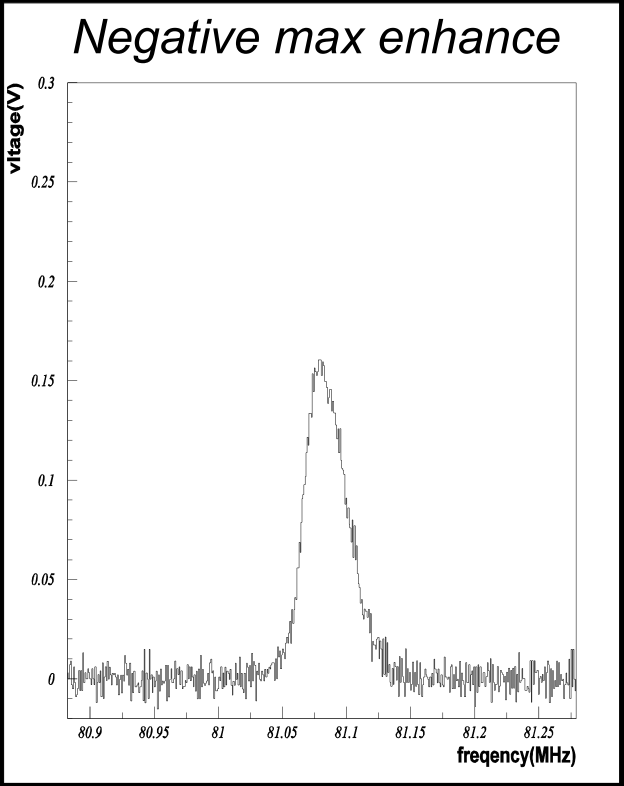
Irradiated microwave frequency:70.072GHz

Positive enhancement S/S_{TE} =3.09

Polarization P_{+}=0.67% (in previous test0.3%)

Negative enhancement by DNP





T=0.90K, B=2.5T,

Irradiated microwave frequency:70.072GHz

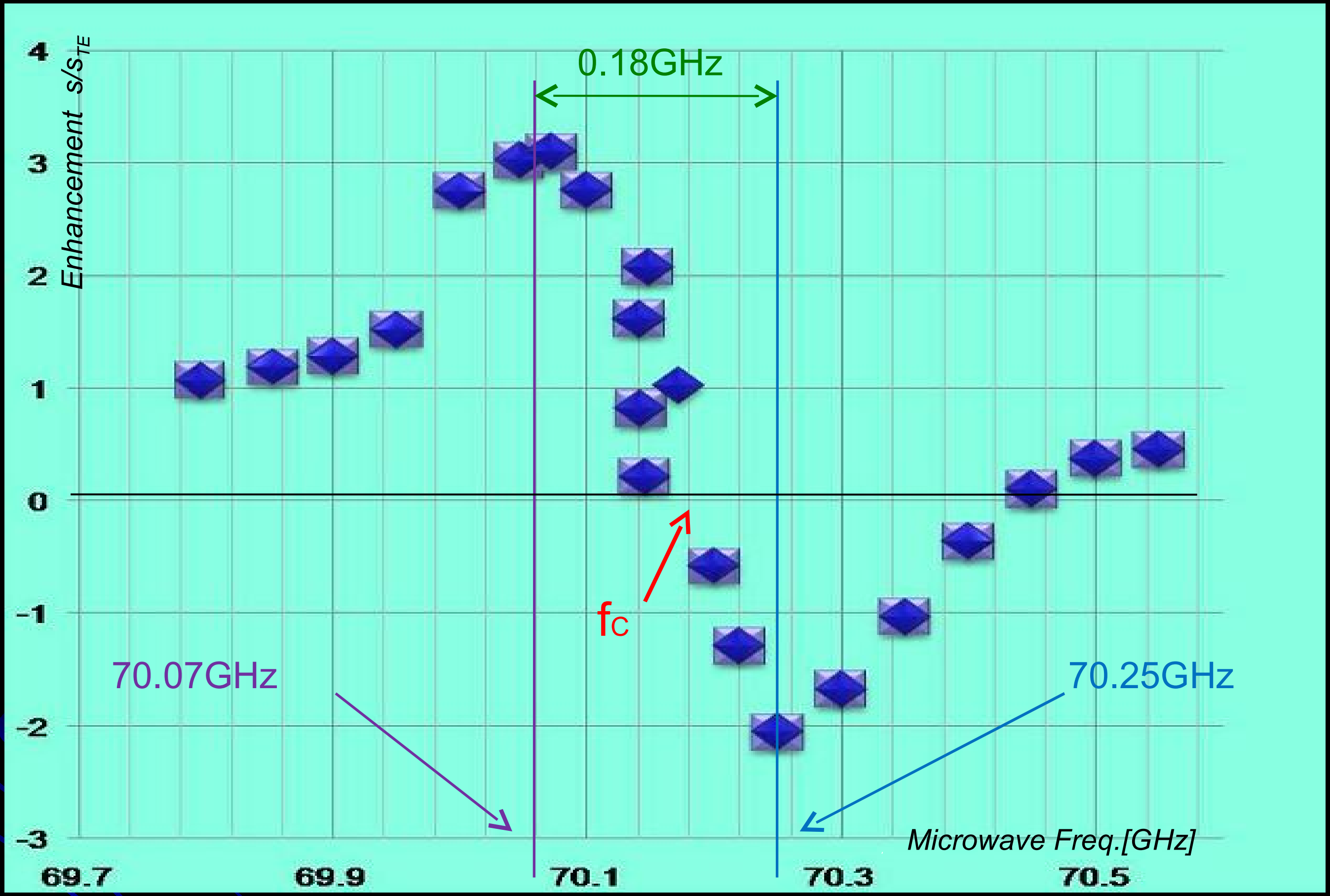
Spin density 0.93×10^{18} [spins/cc]

polarization 0.22

Negative enhancement S/S_{TE} =-2.06

Polarization P_=-0.45% (in previous test 0.21%)

Micro wave frequency dependence



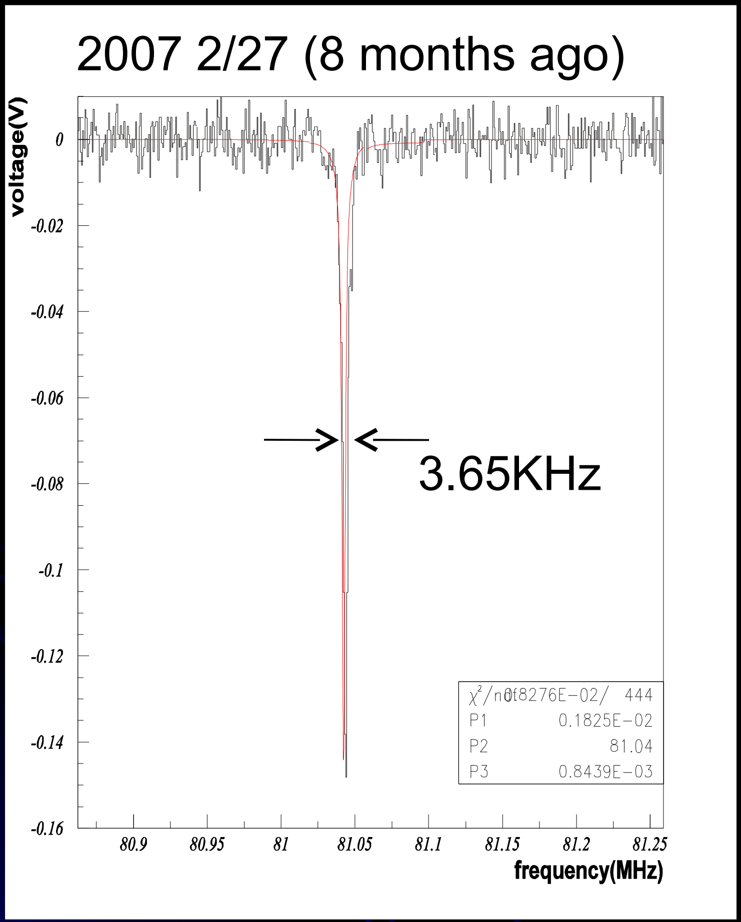
fc:ESR center frequency of TEMPO (=70.177GHz)

●difference between positive max enhancement and negative max enhancement is about 0.18GHz

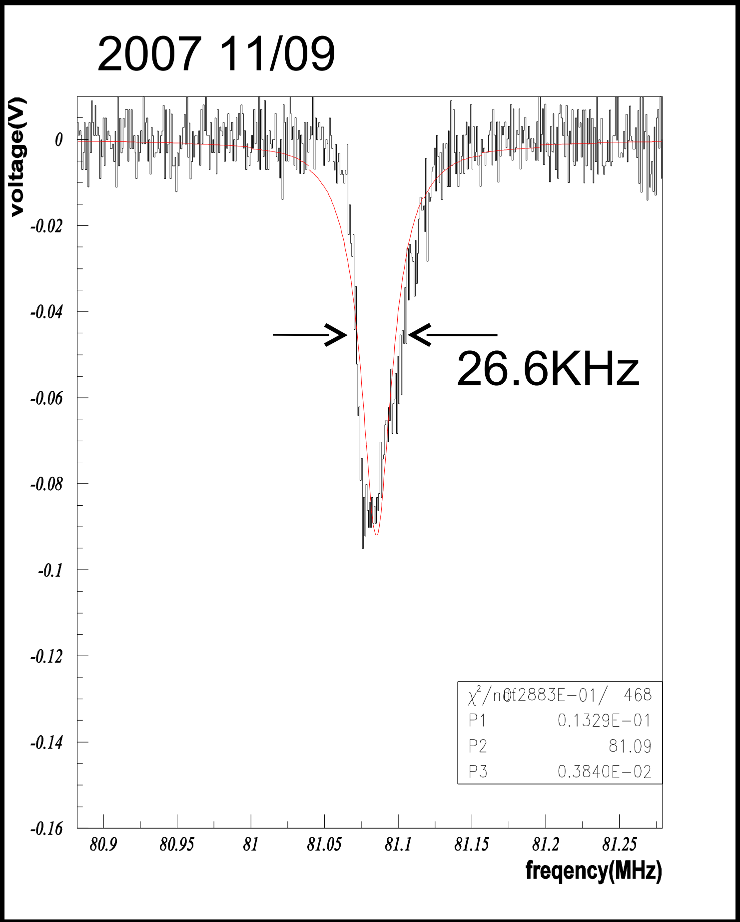
Polarization and enhancement data

	Test of Feburery 27th, 2007	Test of November 9th, 2007
Positive Enhancement S/S_{TE}	2.34	3.09
Negative Enhancement S/S_{TE}	-1.59	-2.06
Positive Polarization	0.30%	0.67%
Negative Polarization	-0.21%	-0.45%

Comparing of TE signal



spin density 4.5×10[18] [spins/cc], T=1.5K, B=2.5T

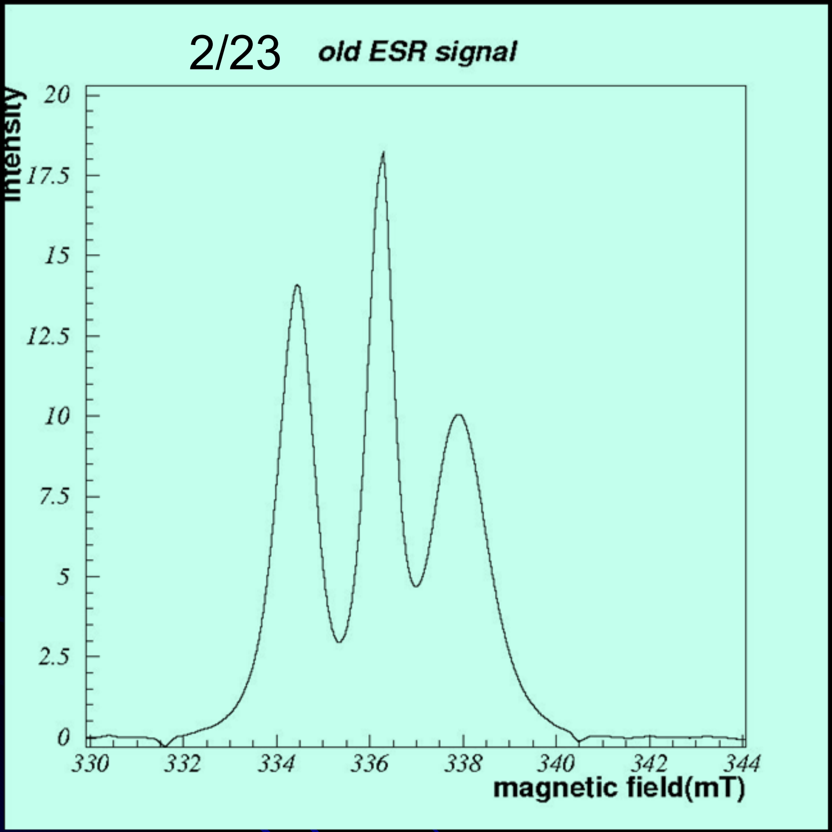


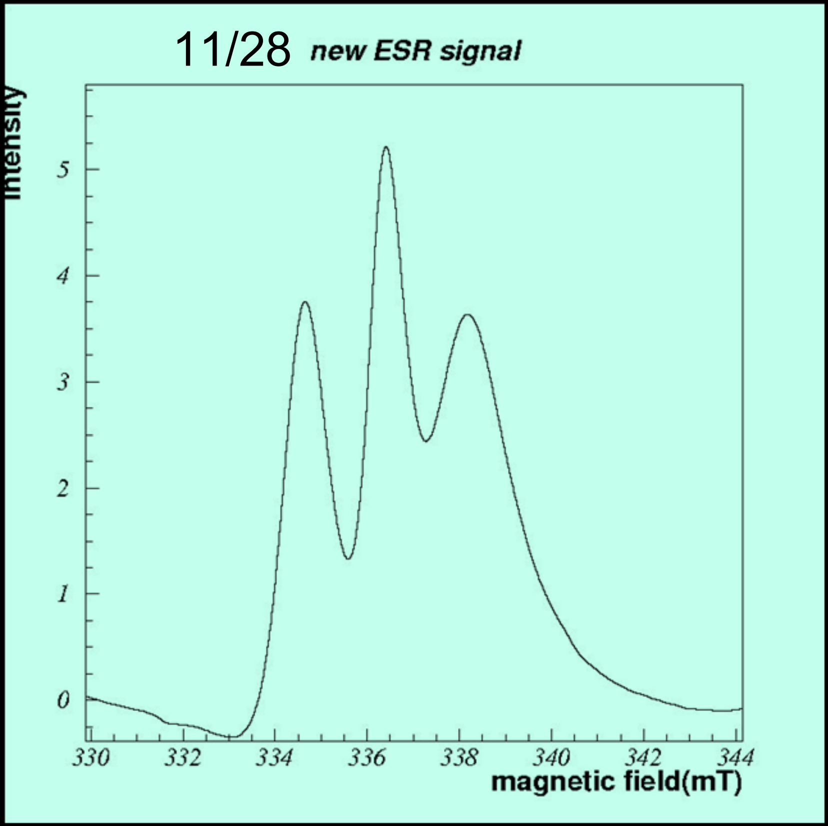
Spin density 0.93×10[18] [spins/cc], T=1.1K,B=2.5T

FWHM=3.65[KHz]

FWHM=26.6[KHz]

Comparing of ESR signal





Spin density: 4.5×10^{18} [spins/cc]

Spin density: 0.93×10^{18} [spins/cc]

- Spin density get into 1/5 compare ESR signal of Febuary 23th.
- Width of ESR signal spreaded

Summary

- We obtained the thermal equilibrium signal of liquid ^3He in zeolite.

  ⇒TE signal width change by spin density and ESR signal width
- We obtained polarization enhancements for liquid ^3He in zeolite by DNP（world record）.
- We obtained different NMR & ESR signal using same zeolite sample.

  ⇒with 8 manth preservation in vacuum, spin density decreased and ESR signal width spreaded

- We should improve experiment systems.
- We should search optimal spin density

Thank you for listening

DNPの原理

e↑ He↑

e↑ He

e↑ He↓

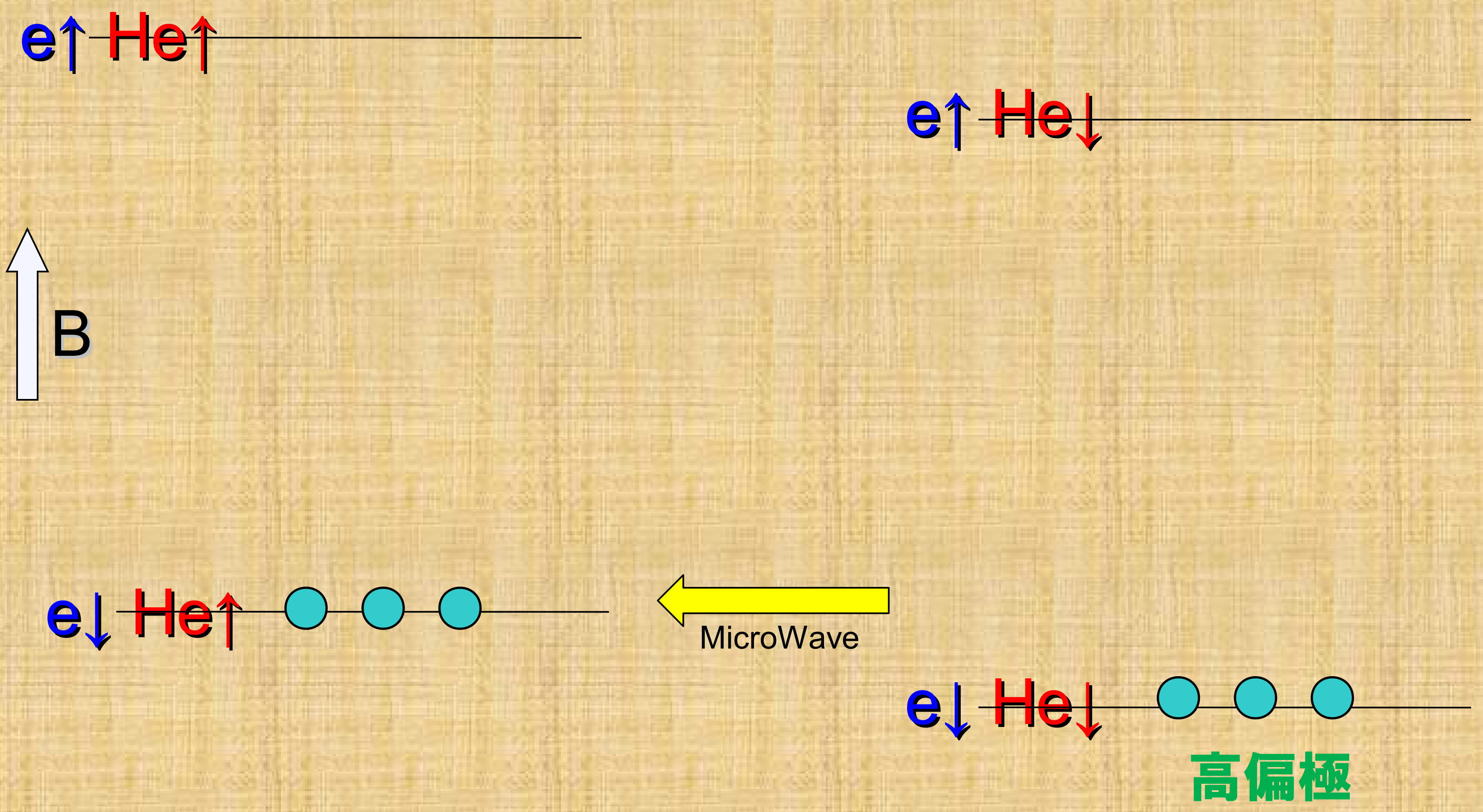
ee

e↓ He↑

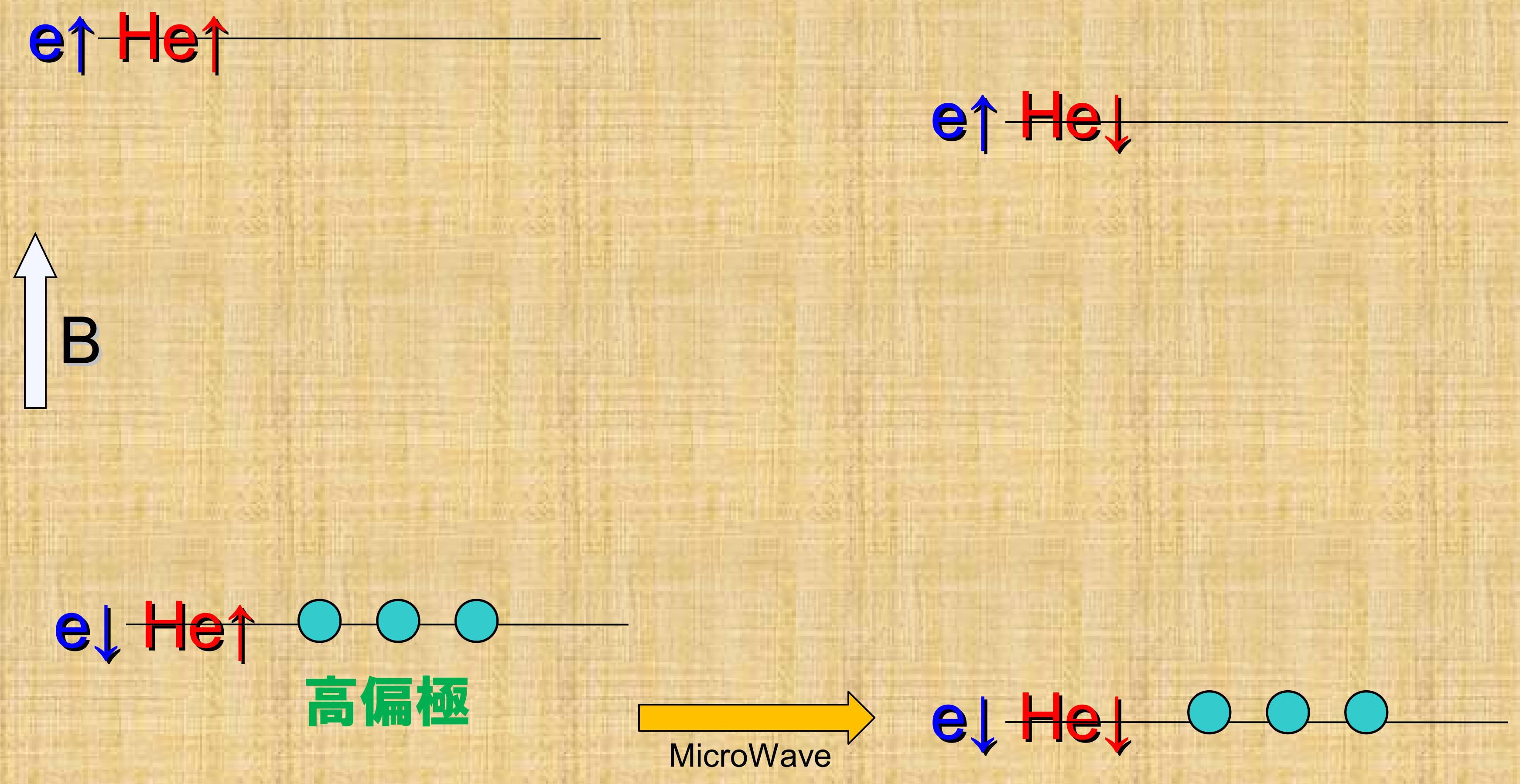
e↓ He

e↓ He↓

DNPの原理

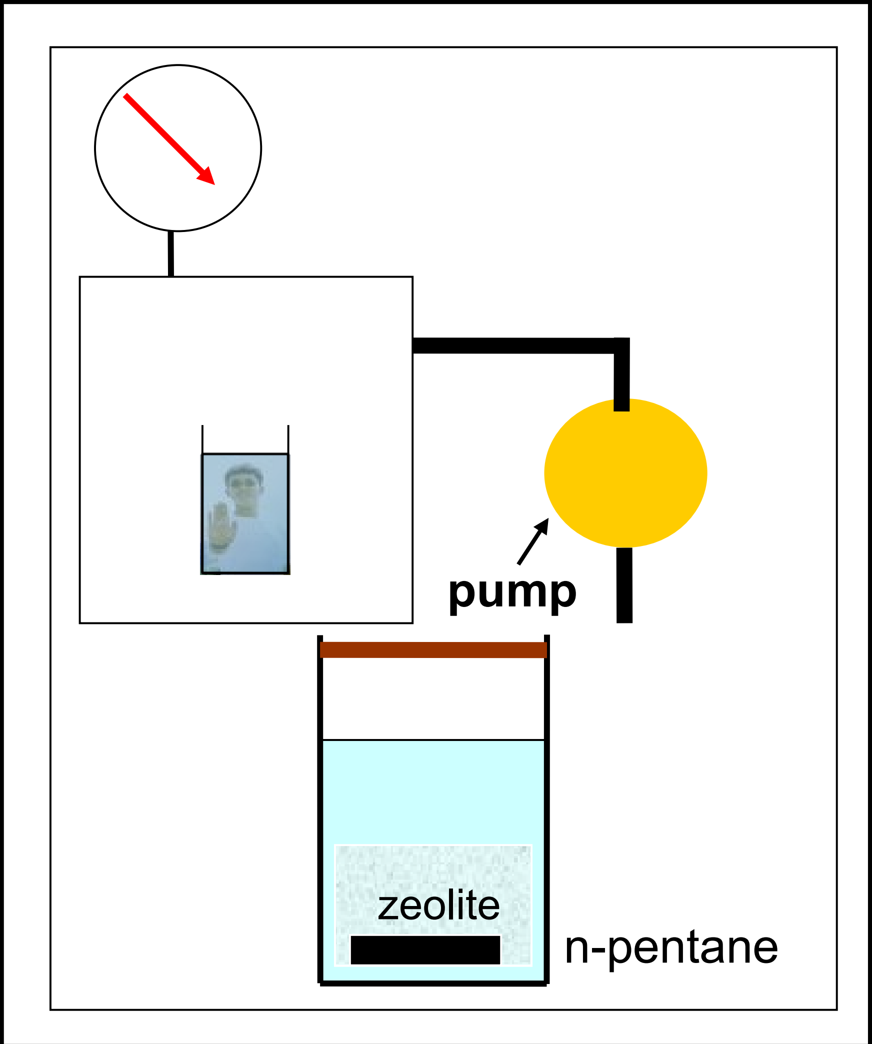


DNPの原理



Zeolite への TEMPO のドープ方法

- TEMPO を n-pentaneに溶す
- zeoliteを加える
- n-pentaneが入った容器を密閉
  8時間攪拌する
- 真空容器に移してポンプで引き
  n-pentanを蒸発させる



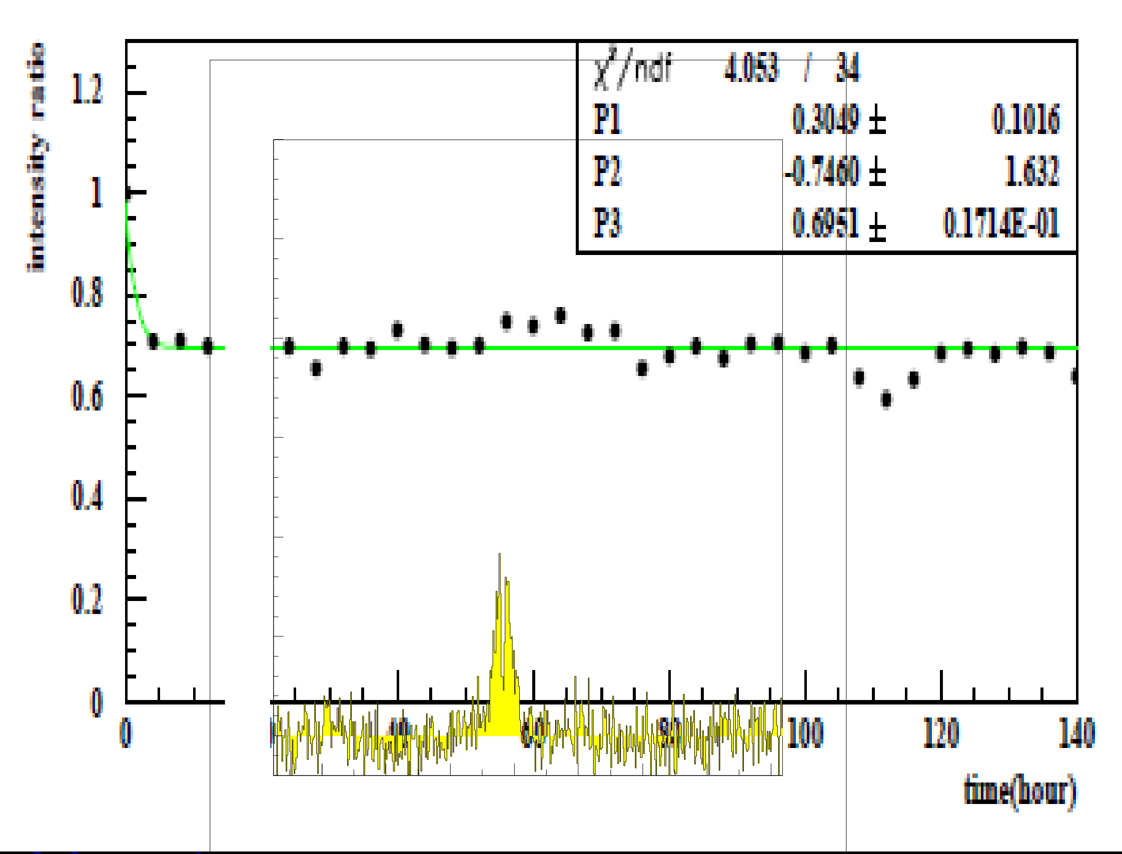
Zeolite and its character

- Mineral which has a micro-porous structure, high hydrophobicity, thermostability and mostly used as a catalyst, ion exchanger, absorber
- ($Na_nAl_nSi_{(192-n)}O_{384}\cdot 240H_2O$(n=48~76))
- Used (HSZ-300serise)TOSOH corporation
  - Cation type: Na
  - SiO_2/Al_2O_3(mol/mol): 5.5
  - Na2O(wt%): 12.5
  - U.C.C. by ASTM :24.63
  - NH3-TPD(mmol/g): -
  - Surface Area(BET, m2/g): 700
  - Crystal Size(μm): 0.3
  - Mean Particle Size(μm): 6

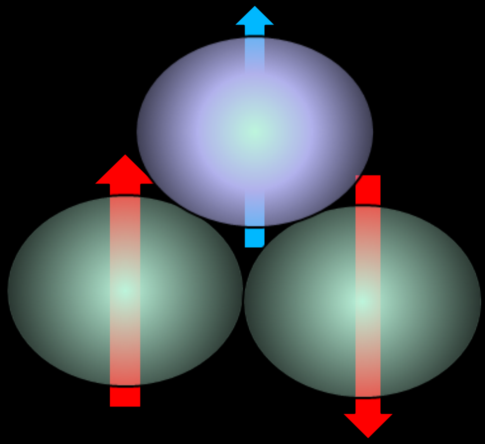
Zeolite's atom

atom	spin	Magnetic moment	Abundance(%)
^{16}O	0	0	99.762
^{17}O	5/2	-1.89371	0.038
^{18}O	0	0	0.2
^{23}Na	3/2	+2.21752	100
^{27}Al	5/2	+3.64141	100
^{28}Si	0	0	92.23
^{29}Si	1/2	-0.55525	4.67
^{30}Si	0	-0.55525	3.10

Decrease of TEMPO in Zeolite



Spin density :7.5×10^{18}spin/cc
Room temperature



偏極度

$$P_{TE} \approx \frac{\mu B}{kT}$$

$$\mu_N = 5.05 \times 10^{-27} J \cdot T^{-1}$$

$$\mu_{He3} = -2.13 \times \mu_N$$

$$k_B = 1.38 \times 10^{-23} J \cdot K^{-1}$$

→0.22%

$$P_V \approx \frac{S}{S_{TE}} P_{TE}$$

$$P_{TE} = 0.22\%$$

$$S = -3.926$$

$$S_+ = -12.141$$

$$S_- = 8.089$$

→P_{V+} =0.67%

→P_{V-} =-0.45%