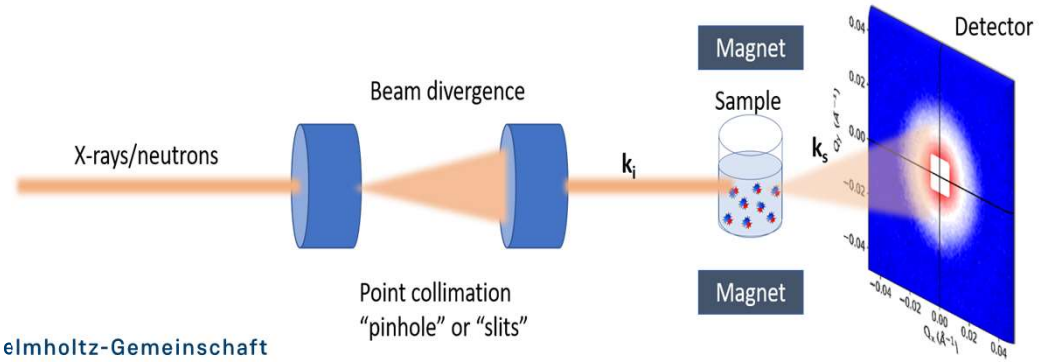
Leibniz Universität Hannover

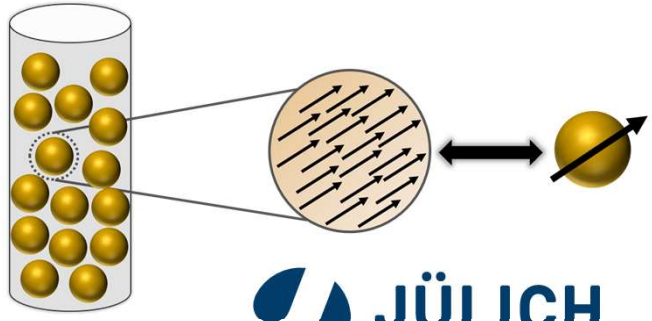
Jülich Centre for Neutron Science

STRUCTURAL AND MAGNETIC CHARACTERIZATION OF IRON-OXIDE NANOPARTICLE DISPERSIONS UPON FREEZING AND MELTING

Maximilian Enneking

Jülich Centre for Neutron Science (JCNS-2) / Forschungszentrum Jülich GmbH / Germany
Leibniz University Hannover

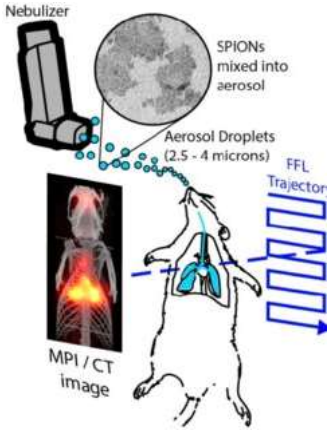




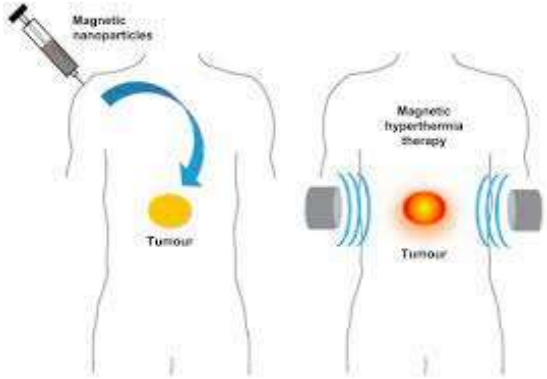
Mitglied der Helmholtz-Gemeinschaft

JÜLICH Forschungszentrum

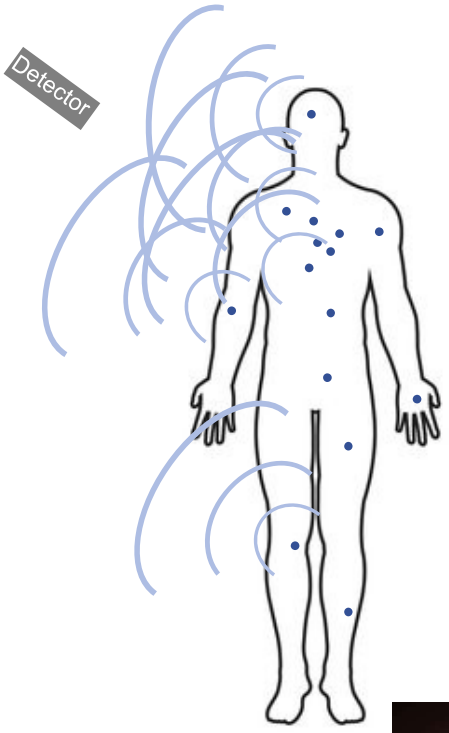
MOTIVATION

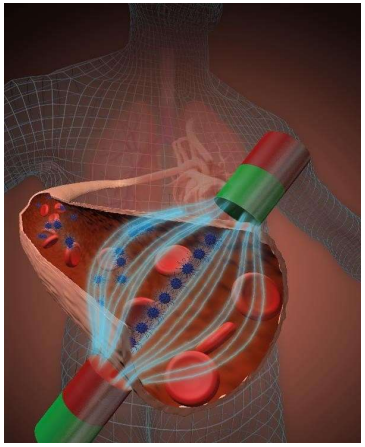


magnetic particle imaging

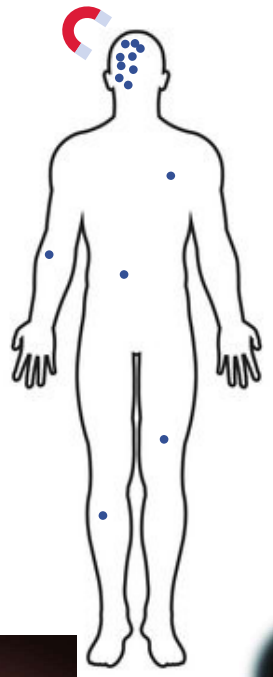


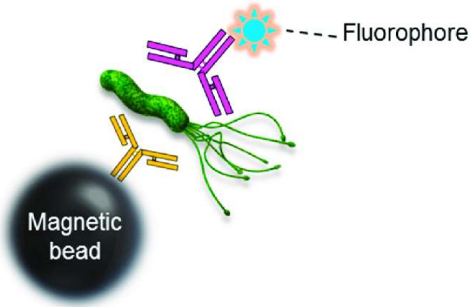
magnetic hyperthermia





targeted drug delivery



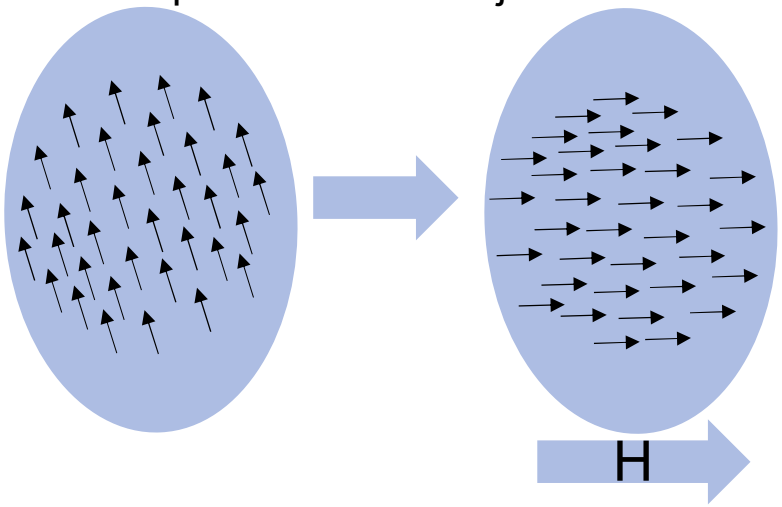


pathogen detection

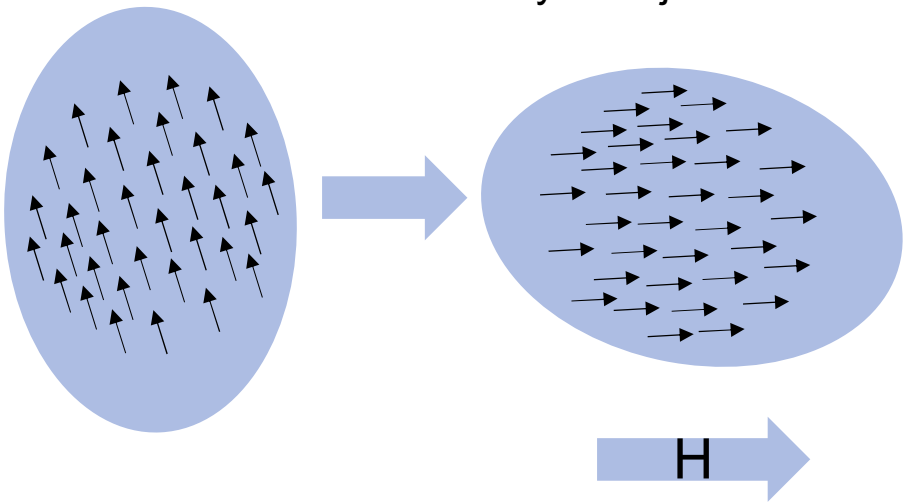
JÜLICH
Forschungszentrum

NÉEL VS BROWN RELAXATION

Néel relaxation:
Spins in nanoparticle turn to adjust to the field

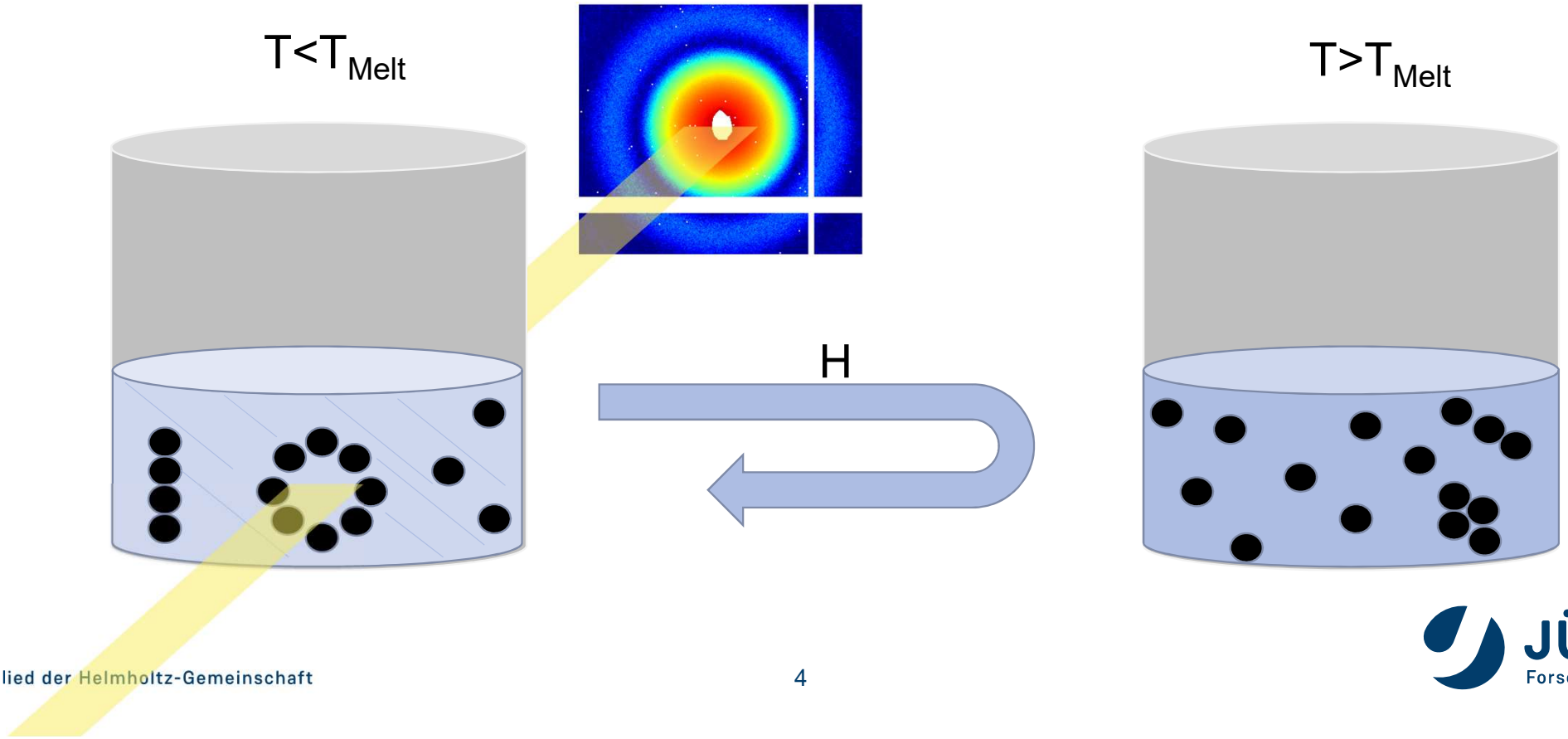


Brown relaxation:
Nanoparticle moves/turns mechanically to adjust to the field



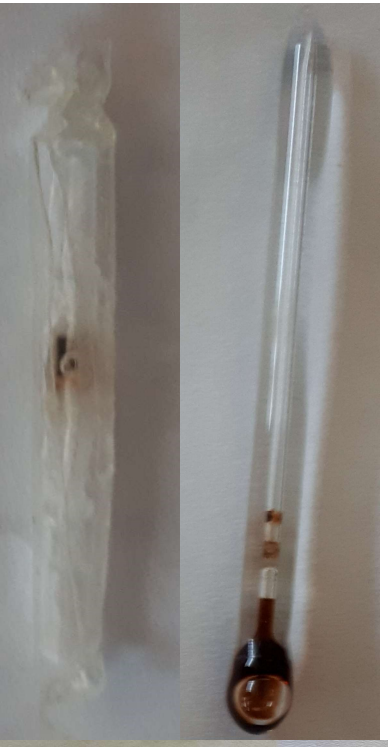
WHAT EXACTLY AM I STUDYING?

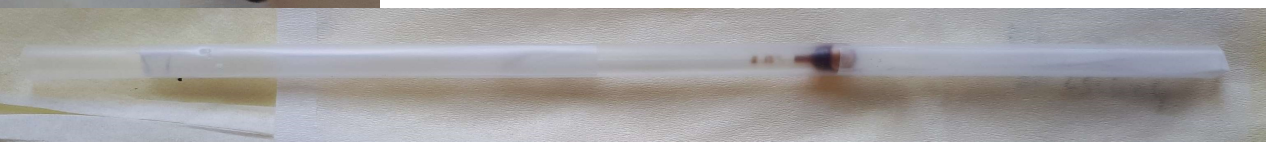
Understanding magnetic and structural properties of FeOx NP dispersions during melting and freezing

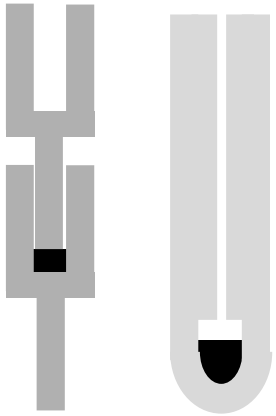


SAMPLE PREPARATION

SQUID







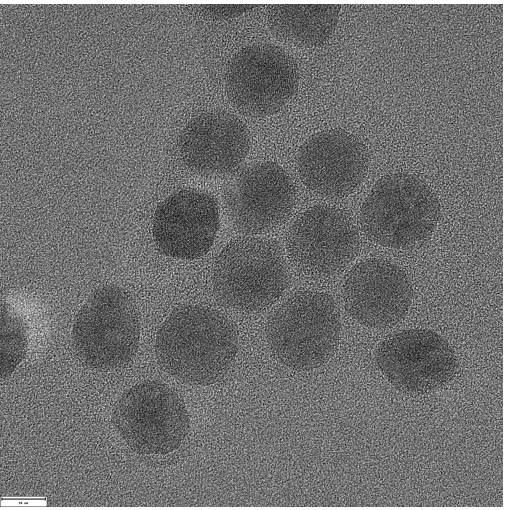
- Spherical FeOx Nanoparticles
- OceanNanotech
- 10 and 15nm
- Dispersed in Water and Toluene

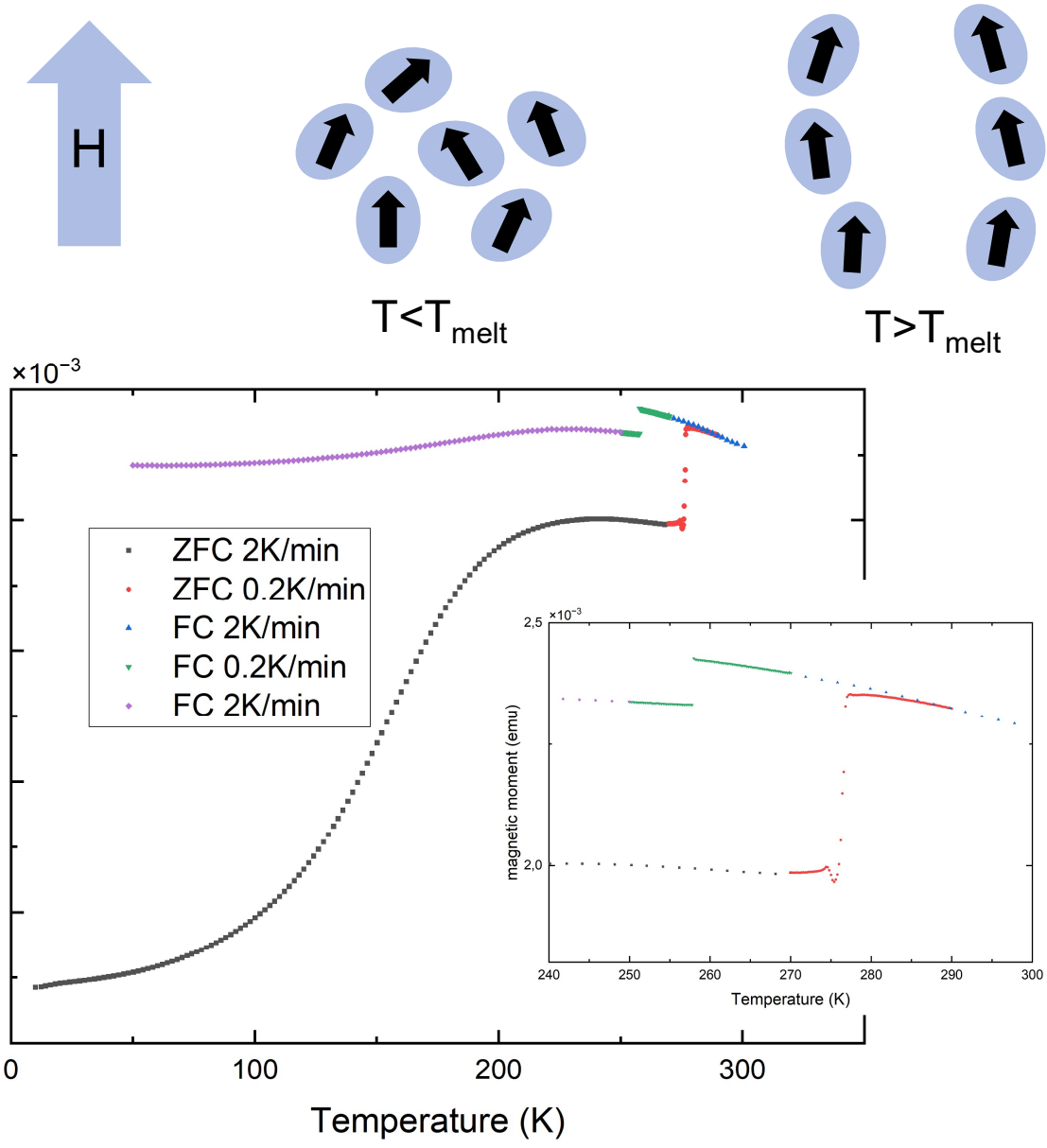
SAXS

15 NM NP IN WATER

Magnetization

- Unfreezing
  - →Brownian relaxation becomes possible
    - →Increase in magnetization
- Freezing
  - →Water supercooles
    - →Gives sudden torque during freezing

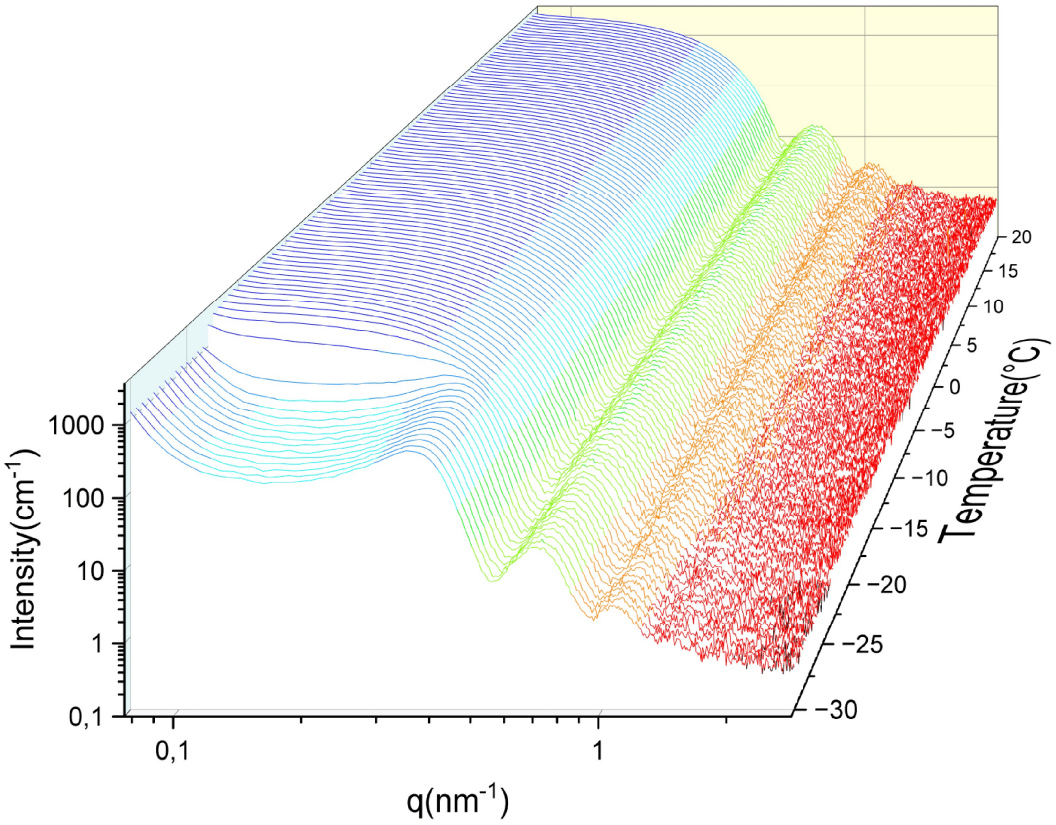




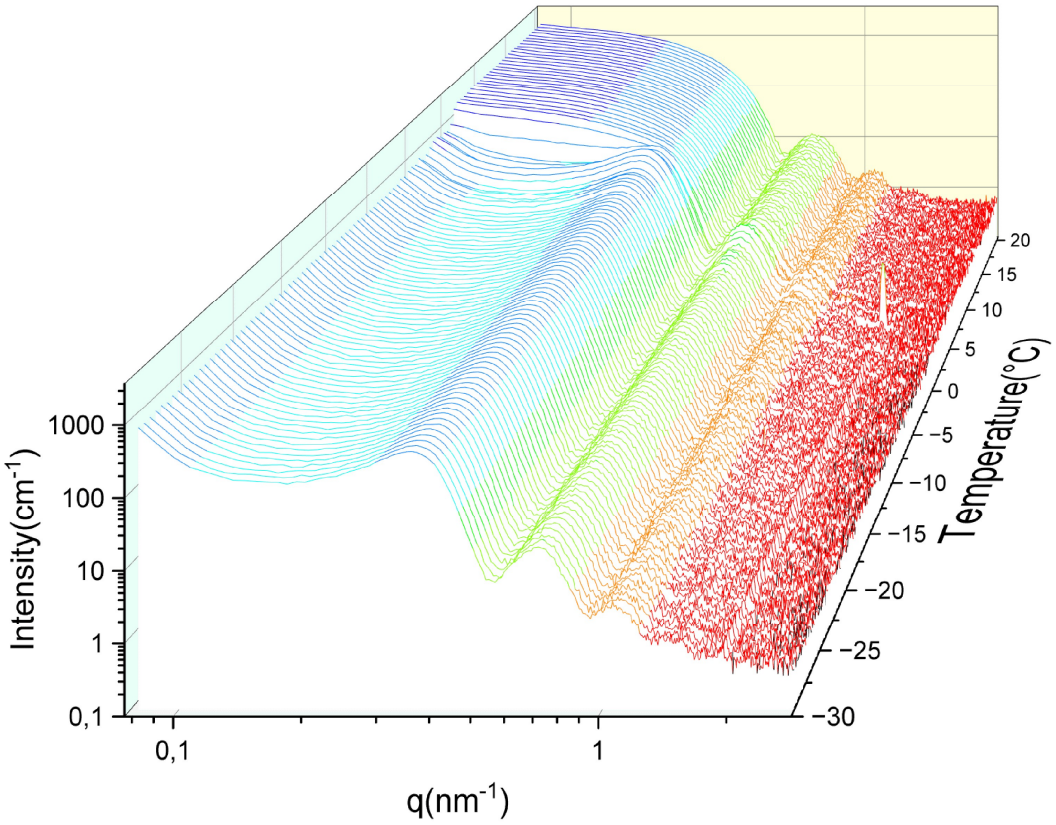
15 NM NP IN WATER

SAXS

SAXS during cooling



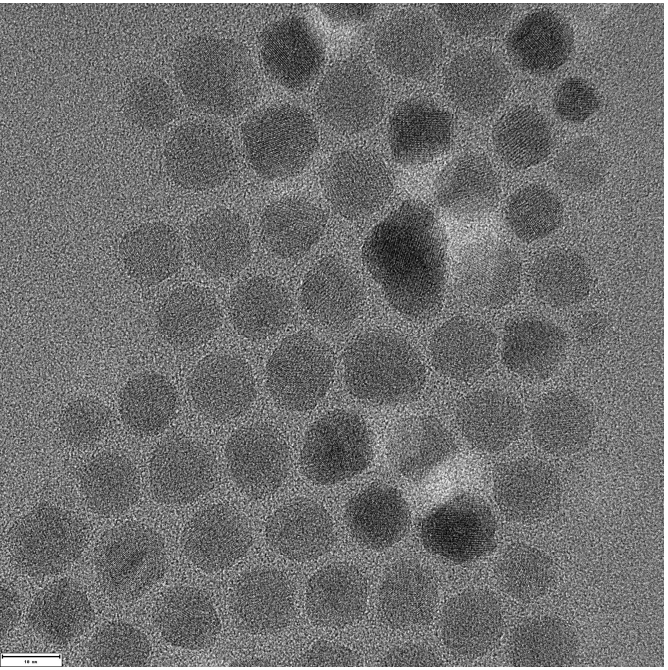
SAXS during heating

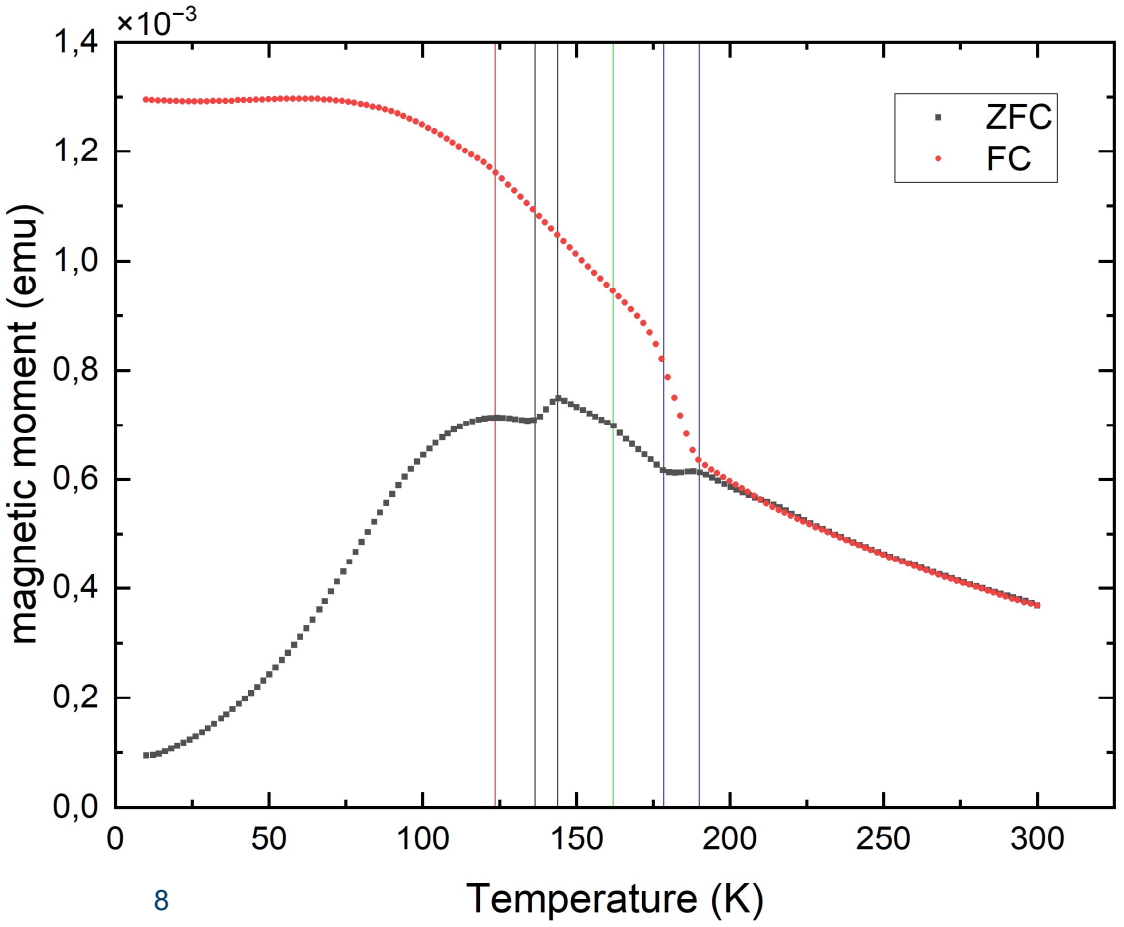


10 NM NP IN TOLUENE

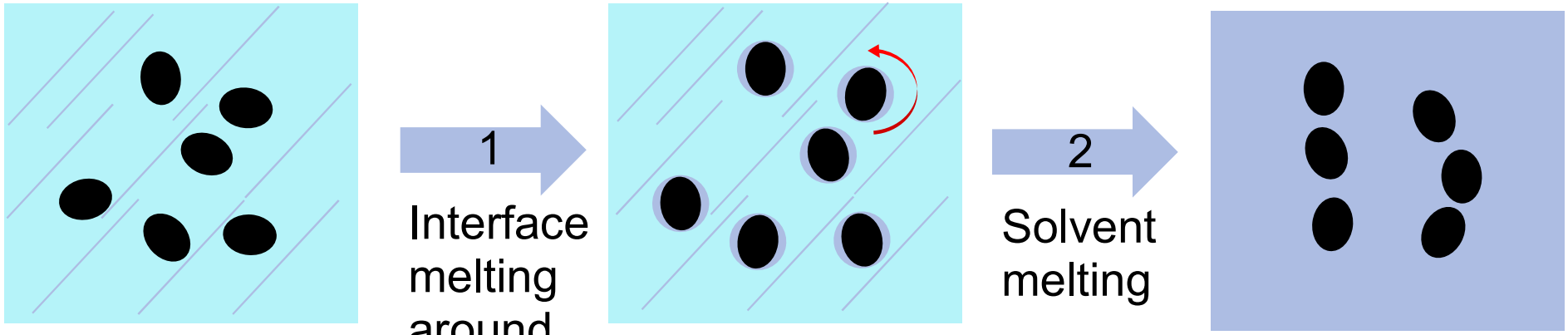
Magnetization

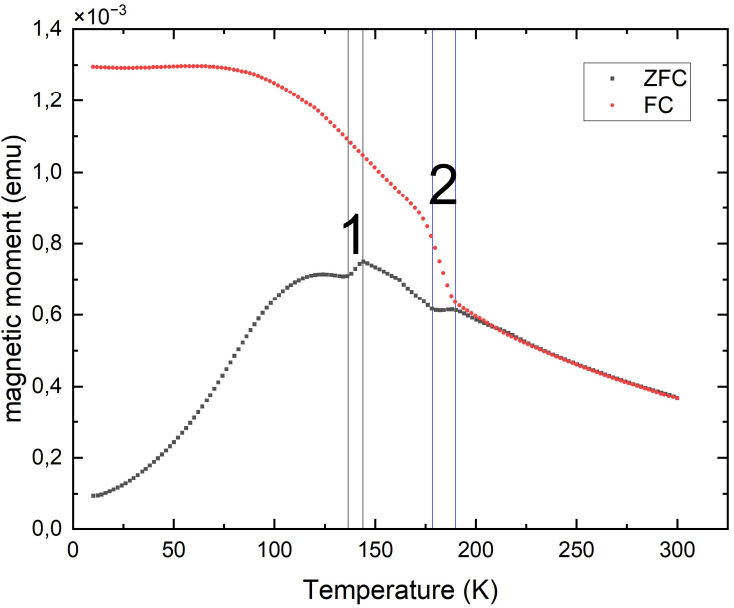
- Red line: Superparamagnetic peak
- Black lines: Suspect NP rotation by interface melting
- Green line: Unknown effect
- Blue lines: Phase transition

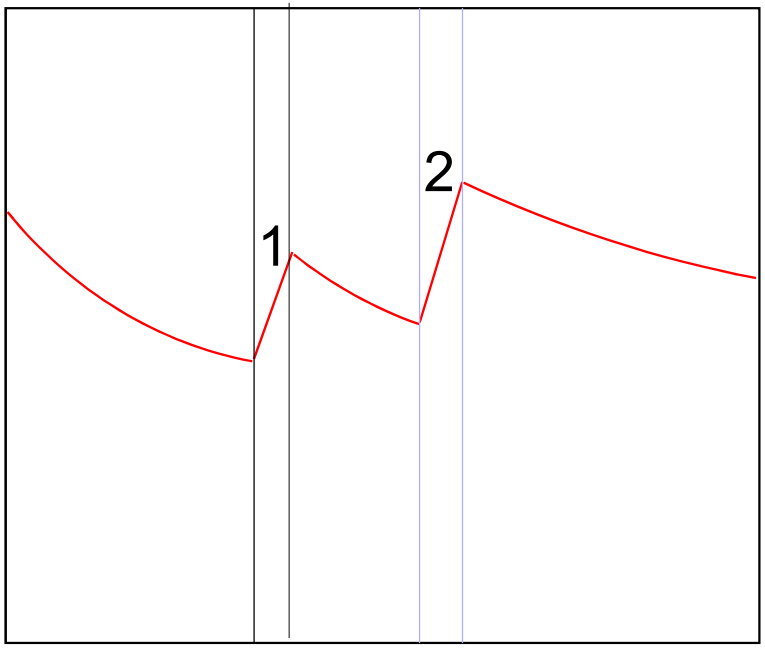




HYPOTHESIS


1
Interface melting around NP
2
Solvent melting

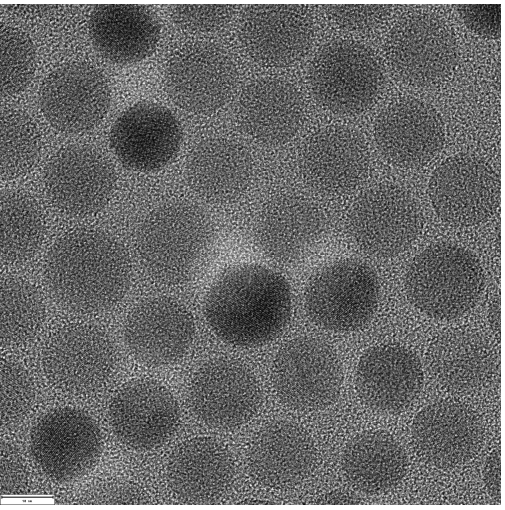

×10⁻³
1,4
1,2
1,0
0,8
0,6
0,4
0,2
0,0
magnetic moment (emu)
ZFC
FC
1
2
0
50
100
150
200
250
300
Temperature (K)

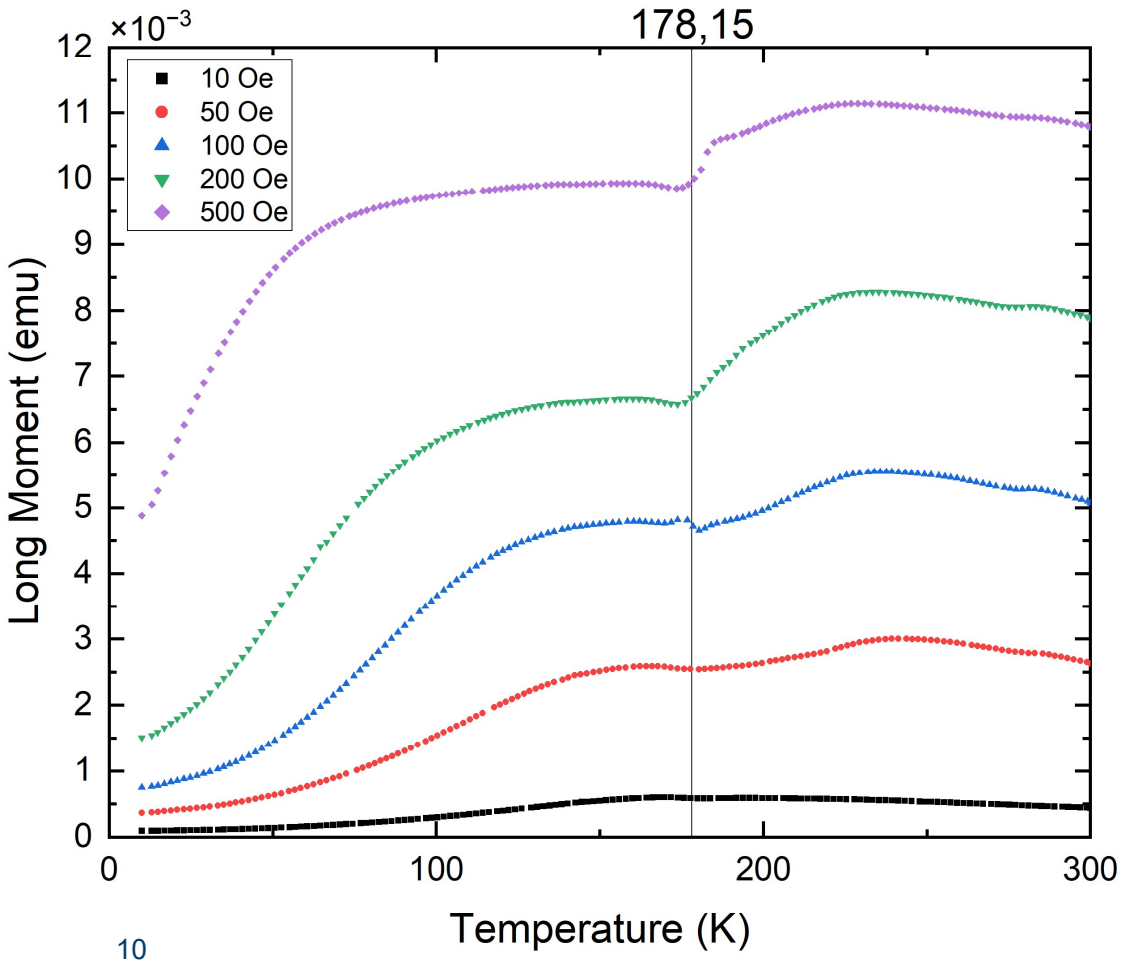

1
2

15 NM NP IN TOLUENE

Magnetization field series

- Néel peak shifts to lower temperatures for higher fields
- Brownian peak above freezing temperature
  → Magnetization shifts at melting point only occurs as Brownian peak shifts to lower temperatures

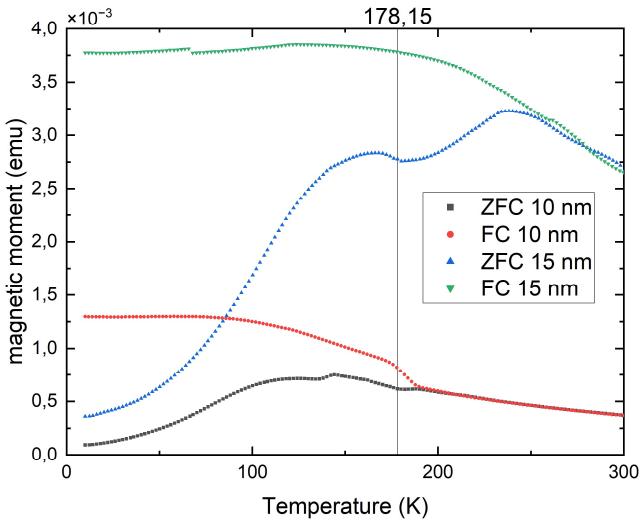


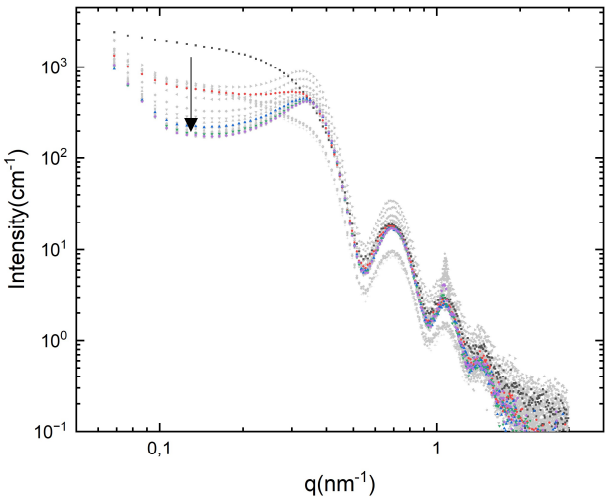


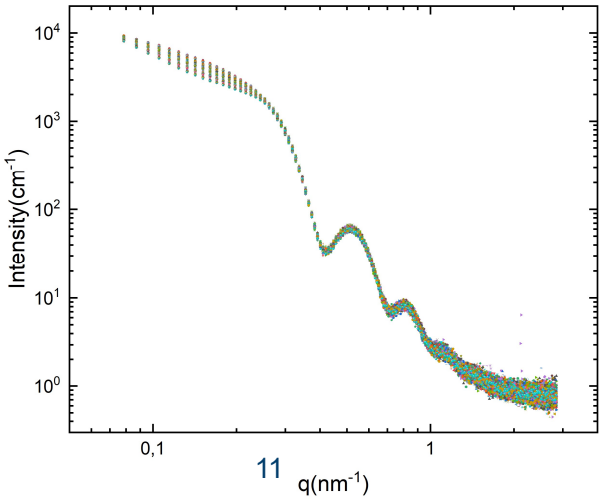
SUMMARY

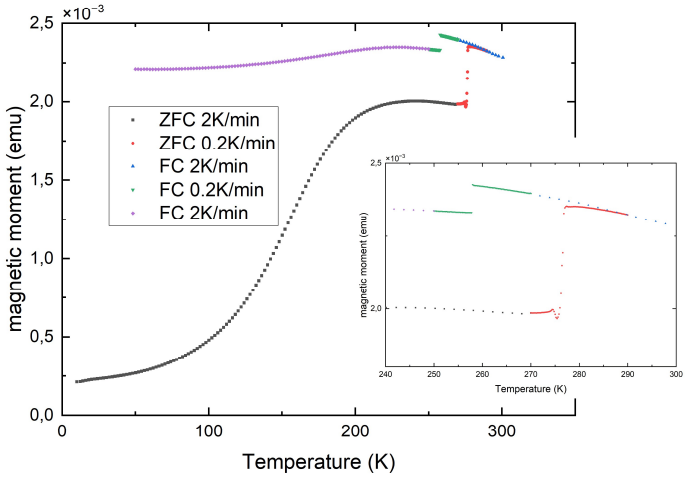
- Brownian relaxation is only possible in liquid phase
  - When the Brownian blocking temperature is below the freezing point

    →Unfreezing is expected to cause a magnetization shift
  - Interface melting may allow partial Brownian relaxation
- Freezing-process causes torque on NP
- NP interaction in frozen state even without field









OUTLOOK

- Cryo-TEM with and without field at ER-C Institute in the Forschungszentrum Jülich
- SAXS with field at GANESHA
- Structural and magnetic characterization for 20 nm particles in water and toluene

ACKNOWLEDGMENTS

- PD Dr. Oleg Petracic
- Dr. Asmaa Qdemat
- Dr. Martin Dulle
- Dr. Johan Buitenhuis
- Prof. Dr. Rolf Haug
- Sascha Ehlert

Jülich Centre for Neutron Science

Institute for Biological Information Processes (IBI-4)

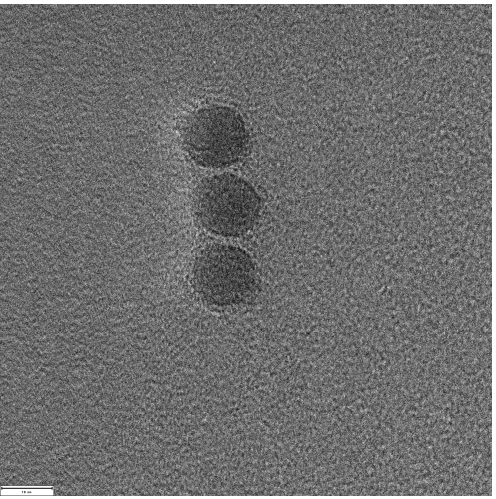
Leibniz Universität Hannover

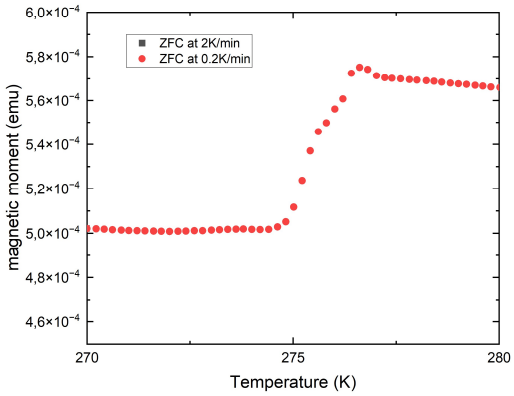
Thank You

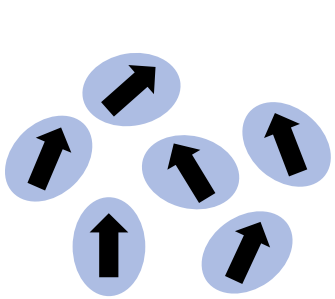
BACKUP: 10 NM NP IN WATER

Magnetization

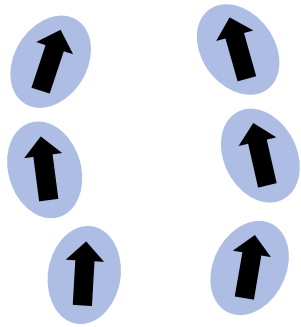
- Unfreezing
  - →Brownian relaxation becomes possible
    - →Increase in magnetization
- Freezing
  - →Water supercooles
    - →Gives sudden torque during freezing



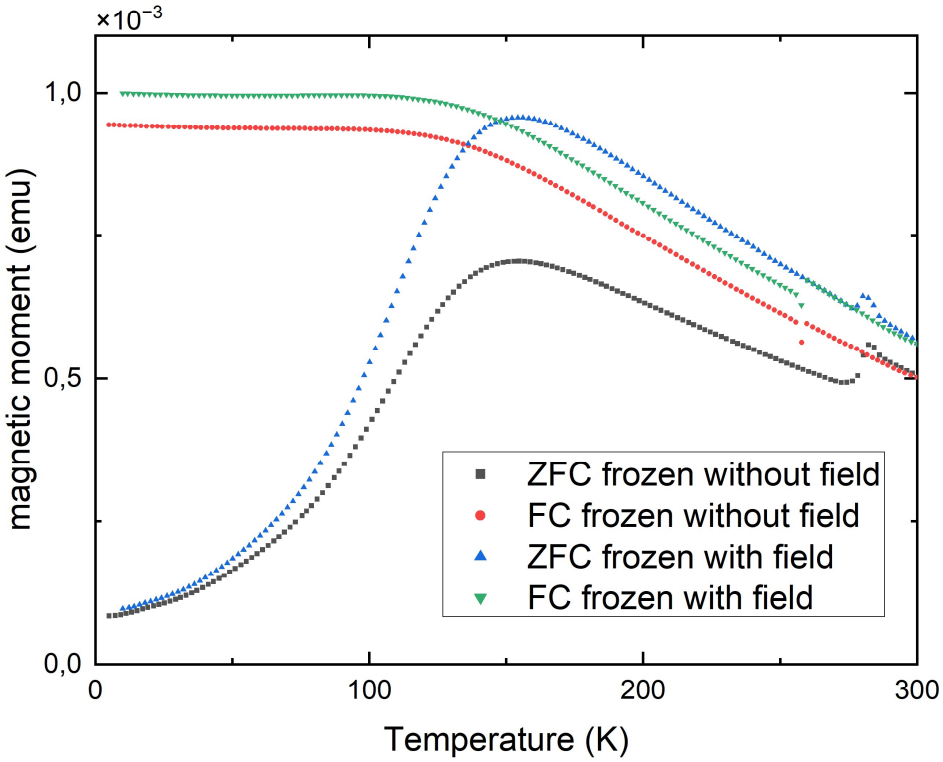




$T<T_{melt}$



$T>T_{melt}$



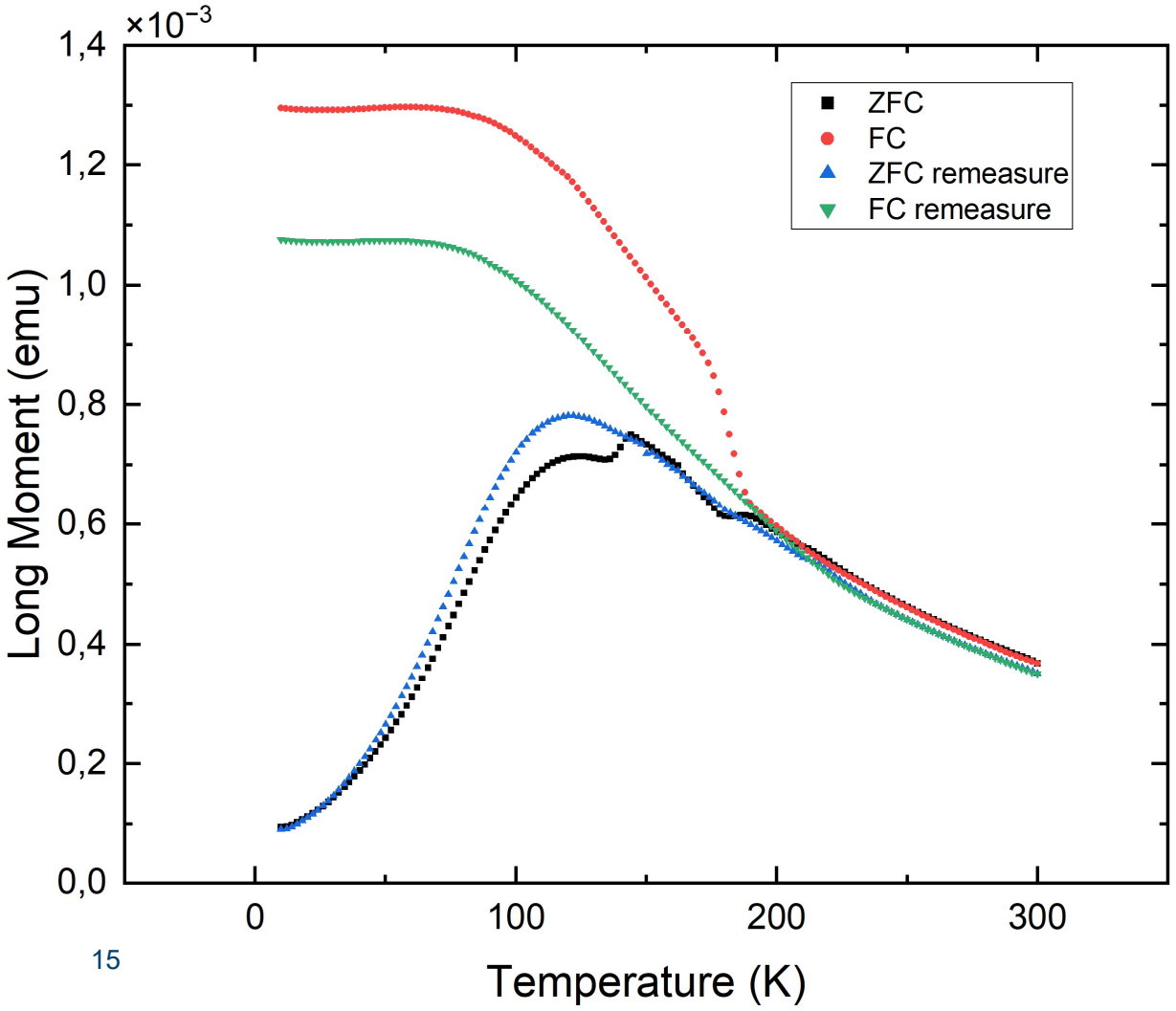
BACKUP: 10 NM NP IN TOLUENE

Magnetization after agglomeration

- Solvent based features disappear
- Néel peak is at roughly the same position

  → Néel-relaxation time is unchanged
- Splitting after Néel peak

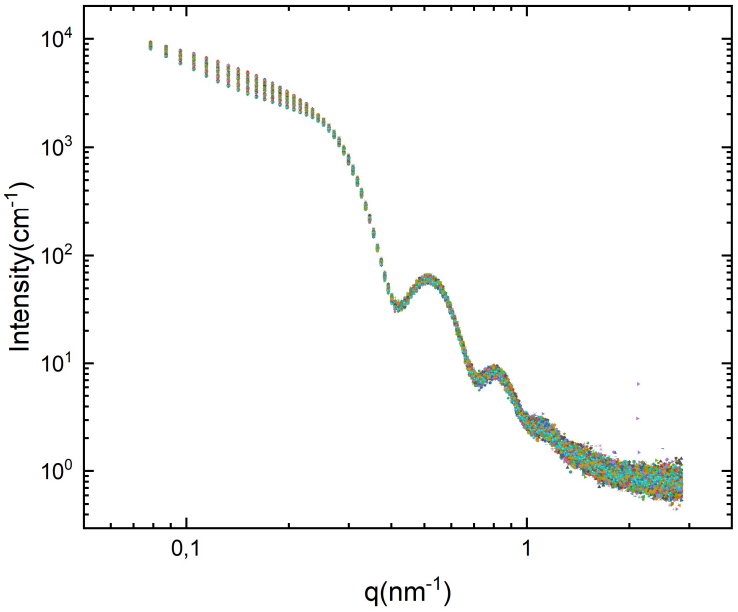
  → High polydispersity



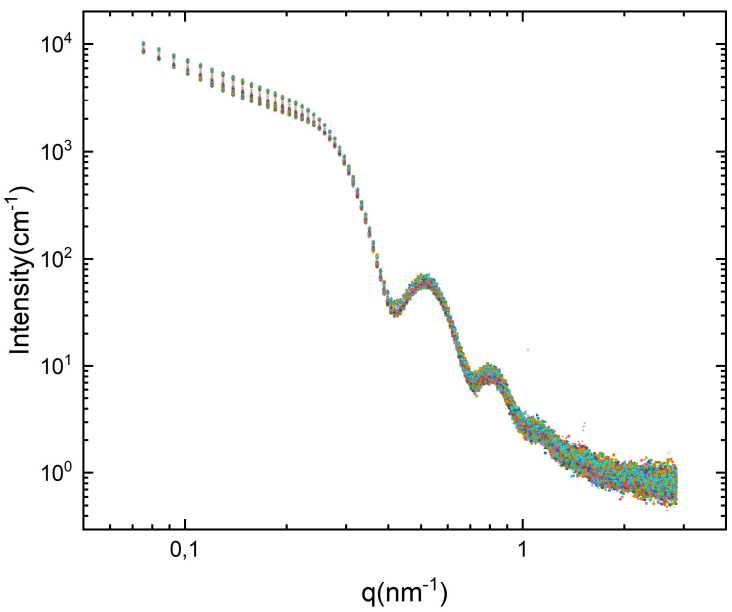
BACKUP: 20 NM NP IN TOLUENE

SAXS

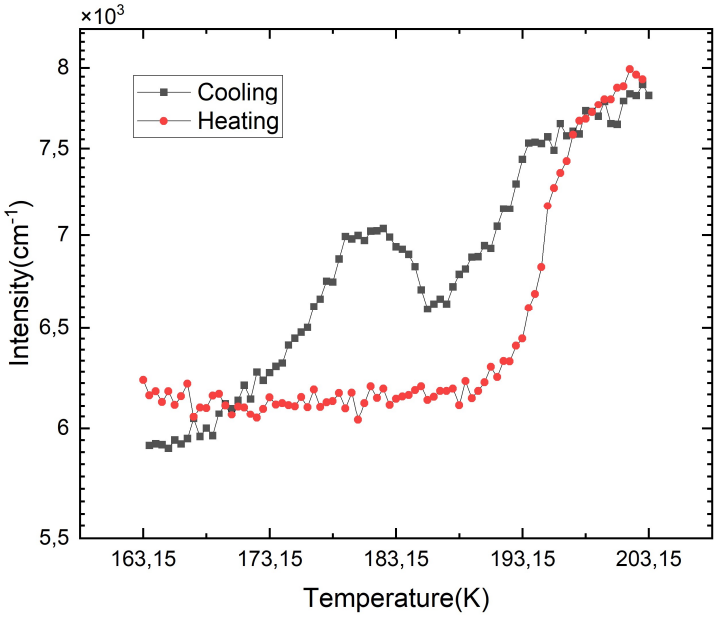
cooling



heating

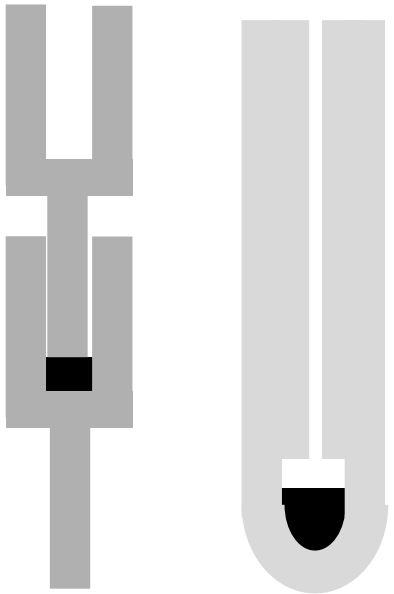


At q=0.096 nm^{-1}



BACKUP:SAMPLE PREPARATION

- Spherical FeOx Nanoparticles
- OceanNanotech
- 10 and 15nm
- Dispersed in Water and Toluene



OUTLINE

- Motivation
- Néel vs Brown relaxation
- What exactly am I studying?
- Water based samples
- Toluene based sample
- Summary