

Nanomagnetism

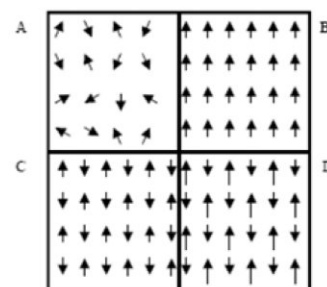
[Physics 3600 - Advanced Physics Lab - Summer](#)

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I. Introduction

Magnetism has been a curiosity for many millennia. According to Chinese writings, magnetite (loadstone, Fe_3O_4) was mentioned about 4000 BCE. Aristotle attributes the first of what could be called a scientific discussion on magnetism to Thales, who lived from about 625 to about 545 BCE. Literary reference to magnetism can be found in a 4th century BCE book called Book of the Devil Valley Master: "The lodestone makes iron come or it attracts it." (Wikipedia) In the 1st century AD, the Greek writer Pliny wrote that magnetite was discovered by the shepherd Magnes, the nails of whose shoes and the tip of whose staff stuck fast in a *magnetick field* he pastured his flocks. Magnetite was mined in the province of Magnesia and the word *magnet* comes from the Greek "magnítis líthos" (μαγνίτις λίθος), which means "magnesian stone".

Magnetite is an example of just one type of magnetism. Although magnetite can be polarized to generate a permanent magnetic dipole, it is actually classified as a **ferrimagnet** rather than a **ferromagnet**. The table lists six classifications of magnetism. The last one on the list is superparamagnetism, and is the subject of this laboratory experiment.



Class	Critical Temperature	Magnitude χ	Structure
Diamagnetic (Al, Cu, other metals)	none	weak $\sim 10^{-6} \text{ B } 10^{-5}$	No permanent dipole moment - See A
Paramagnetic (Cu doped with Fe)	none	moderate $\sim 10^{-5} \text{ B } 10^{-3}$	No permanent dipole moment Dipoles do not interact
Antiferromagnetic (MnF_2)	Neel T_N	moderate $\sim 10^{-5} \text{ B } 10^{-3}$	Antiparallel dipole moments - See C
Ferrimagnetic (Fe_3O_4 , CrO_2)	Curie T_c	strong $> 10^{-3}$	Permanent dipole moment (unequal antiparallel) - See D
Ferromagnetic (Fe, Ni, Co)	Curie T_c	strong $> 10^{-3}$	Permanent dipole moment (parallel moments) - See B
Superparamagnetic (nanoparticles)	Blocking T_B	strong $> 10^{-3}$	Permanent dipole moment below T_B

Superparamagnetism occurs when a ferri/ferromagnetic material is reduced in size below about 50 nm in the largest dimension. At any temperature, all the magnetic ions in the nanoparticle ($\sim 10^4$) are locked together and produce a large permanent magnetic dipole moment. However, for temperatures higher than a so-called Ablocking@ temperature, T_B , the moment flips 180 degrees very rapidly and all you see is the resulting average moment which is near zero. Only for temperatures below T_B does the moment stay fixed in one direction during the time of the measurement. Thus the particles exhibit a ferromagnetic-like response for temperatures below T_B , but possess a paramagnetic-like response above T_B . The Asuper@ part of superparamagnetism arises from the large magnetic moment of the entire particle which

aligns in the field, whereas in normal paramagnetism only the small moments of single ions are aligning in the field.

In this lab experiment you will measure the magnetic properties of superparamagnetic nanoparticles in a state-of-the-art SQUID magnetometer. The magnetite (Fe_3O_4) magnetic nanoparticles have an average diameter, and a distribution in the diameters. You will measure the magnetic moment as a function of applied magnetic field and temperature. By modeling these dependencies you will determine the average diameter of the magnetic nanoparticles

II. Apparatus

Fe_3O_4 (magnetite) magnetic nanoparticle sample mounted in straw holder, 050508-db
[SQUID Magnetometer \(Quantum Design, Inc.\)](#), located in Egan 362
 - operating instructions in Appendix A

III. Procedure

A. Magnetic Moment versus Applied Magnetic Field at Room Temperature

Measure the **magnetic moment** as a function of the applied magnetic field, $m(H)$.

At $T=300$ K, measure $m(H)$ for about 20 magnetic fields from $H=3000$ Oe to -3000 Oe.

Write a sequence (program) to measure $m(H)$.

Have it checked by the instructor.

B. Magnetic Moment versus Temperature for ZFC and FC Conditions

Zero Field Cooling (ZFC) -

In the first part of the experiment, the sample temperature is lowered to $T=10$ K without applying a magnetic field. Thus, it is call the ZFC condition. At the lowest temperature, a small magnetic field, $H=100$ Oe, is applied. The magnetic moment of the sample is measured for increasing temperature, $m(T)$.

Field Cooling (FC) -

In this part of the experiment, the sample temperature is lowered to $T=10$ K from $T=300$ K in a small field of $H=100$ Oe. Thus, it is call the FC condition. Again the magnetic moment of the sample is measured for increasing temperature.

Write sequences to:

(1) measure the ZFC moment for temperature **increasing** from $T=10$ K to 300 K;

(2) measure the FC moment for temperature **increasing** from $T=10$ K to 300 K.

Put all 3 sequences, $m(H)$, ZFC and FC, into one sequence.

Have it checked by the instructor and include it in your report.

C. Collect Data

Insert the sample into the magnetometer. Center the sample.
Run the sequence to collect data for $m(H)$ and $m(T)$.

Plot $m(H)$.

Plot $m(T)$ for both ZFC and FC conditions on the same graph.

What is the blocking temperature, T_B (peak in the ZFC data)?

D. Determine the Average Particle Diameter from $m(H)$

The magnetic (dipole) moment of a nanoparticle can be approximated by the Langevin function, $L(z)$,

$$m(H) = m_o L(z),$$

$$L(z) = 1/\tanh(z) - 1/z.$$

Here, $z = \mu H/k_B T$ is the ratio of magnetic energy to thermal energy, $\mu = n\mu_B$ is the magnetic (dipole) moment of a particle, $\mu_B = 9.27\text{E-}21$ erg/gauss is the Bohr magneton, n is the average number of Bohr magnetons in the particle, $k_B = 1.38\text{E-}16$ erg/K is the Boltzmann constant, and T is the temperature. Note that m_o is a simple scaling parameter which is not of interest here.

The *magnetization* of the particle is the magnetic moment per unit volume, $M = \mu/V = n\mu_B/V$. Assume that $M = 400 \nabla 10$ emu/cm³ is the saturation moment of Fe₃O₄, and the sphere volume V .

Fit $m(H)$ to a Langevin function to determine the average number of Bohr magnetons, n .
Determine the average particle diameter from n , and the uncertainty.

E. Determine the Average Particle Diameter from the Blocking Temperature

Thermal blocking occurs when the thermal energy, $k_B T$, is less than 1/25 of the so-called anisotropy energy, $K_{\text{eff}} V$. Thus, at the blocking temperature the ratio of anisotropy energy to thermal energy is given by

$$K_{\text{eff}} V / k_B T_B = 25.$$

Here, the effective anisotropy constant for Fe₃O₄ is $K_{\text{eff}} = 1.0 \pm 0.2 \text{ E6 erg/cm}^3$.

Determine the average particle D from T_B , and the uncertainty?

F. Summary

In a table, compare the values for D, including uncertainties. Discuss.
In the Introduction, summarize **superparamagnetism**.

Optional: Determine the distribution in diameter from the ZFC curve.

Appendix A - Instructions for SQUID Magnetometer

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The SQUID magnetometer, Quantum Design MPMS, is located in Egan 430. You can get a key to enter the room from Egan 361. For questions, first ask other students who have done the experiment.

The sample of magnetic nanoparticles is mounted in a plastic straw, labeled 050508-db, which can be found on the black table located to the left of the magnetometer. Someone will show you how to mount a sample on the long sample rod and then insert it into the magnetometer.

INSTALL SAMPLE

- (1) Confirm that the sample temperature is 300K
- (2) Mouse click on the Remove/Install button in computer program
- (3) Remove sample only after you see the **yellow** light come on:
 - Remove the black stopper from the top of the sample chamber.
 - Note: do not leave the black cap off the top for more than one minute.*
 - If there is a sample rod already in (as observed by a blue metallic part visible at the top of the sample tube) insert the white screw one turn, then lift the blue metal part rod a few cm out of the sample tube.
 - Unscrew the black stopper, then slowly remove the long sample rod completely out.
 - Replace black cap.
- (4) Mount Sample: mount your straw with the sample onto the black sample rod; place a small black plug on the bottom
- (5) Put the removed sample in its labeled paper straw-cover, which should be on the top of the magnetometer
- (6) Place your labeled paper straw-cover on top of the magnetometer
- (7) Install sample:
 - Slowly lower the sample rod with your sample straw attached. Do not drop the rod when near the end.
 - When nearly fully inserted, insert the white screw one turn, then push the rod down gently
 - Remove screw, then use the end of the screw to gently push down on the blue metal
 - Reinstall the black plug (screw up) on the top of the sample chamber
- (8) Record your name, sample, and helium level in the log book

DO NOT
open the top of the sample chamber
unless the temperature is 300K

SOFTWARE CONTROL

The next processes are done via the computer.

- (1) Click the Purge Sample button
- (2) Then click the Finished button
- (3) Type in the name of your sample; click OK

CENTER THE SAMPLE

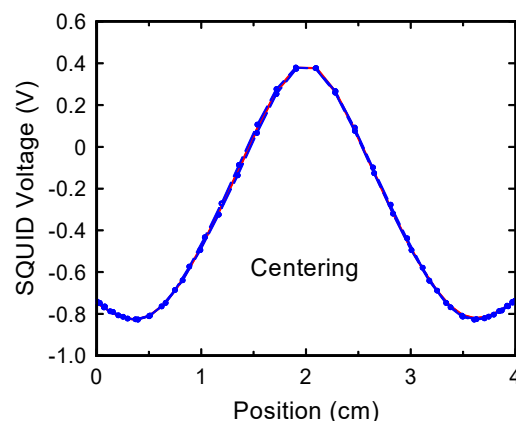
- (1) Click Target Field at the bottom to set field to $H=100$ Oe
- (2) Click Center /RSO at the top; then click Center; a graph will soon appear
- (3) If observed maxima is not within 0.3cm of 2cm then
 click Adjust Position/Adjust Automatically
- (4) Close window when observed minima is near 2.0cm as shown here

COLLECT DATA

- (1) Write a sequence to measure three things:
 - (i) $m(H)$ at $T=300$ K
 - (ii) $m(T)$ for ZFC from $T=10$ to 300 K
 - (iii) $m(T)$ for FC from $T=10$ to 300 K
- (2) Have instructor check the sequences
- (3) Run Sequence

SAVE DATA IN SPREADSHEET FORMAT

- (1) Open the file "ExportData.exe"
- (2) Load data file "xxxxx.rso.dat"; input file name into "Export File" (different from loaded file name)
- (3) Select "Select Data": click on temperature, field, moment



(4) Select "Export Data"