EXPERIMENT 1

PURPOSE

To determine experimentally the optimum mole ratio between $FeCl_2$ and $FeCl_3$ reactants used in the synthesis of a so-called "ferrofluid" whose appearance is altered by the presence of a magnet.

INTRODUCTION

The successful synthesis of a ferrofluid begins with the formation of a *colloidal suspension* of magnetic nanoparticles in a liquid medium. These colloids are suspensions of very small particles on the order of 100 nanometers (10nm) in size. By synthesizing the nanoparticles in the presence of molecules called surfactants, the particles can be suspended in solution rather than clumped together.

These ferrofluids possess the interesting property of "spiking" that you observed in Investigation 1. This spiking will be used to determine whether a high-quality ferrofluid has been produced , conversely, the formation of a high-quality ferrofluid will be used to optimize the synthesis and to estimate the stoichiometric ratio needed for complete reaction of the two iron solutions used in the synthesis.

You and your partner will make an aqueous-based ferrofluid following the procedure below. Your instructor will assign to you a specific combination of the $FeCl_2$ and $FeCl_3$ solutions and your results will be pooled with those from your classmates.

SAFETY PRECAUTIONS

All of the chemicals used in this synthesis are to be handled with extreme caution. Remember that ferrofluids can be messy and this particular one will permanently stain almost any fabric. Do not let the fluid come into contact with any magnet. Keep the magnet and the ferrofluid well separated at all times.

PROCEDURE

1. To a 100 mL beaker add the amounts of the stock 2M $FeCl_2$ and stock 1M $FeCl_3$ solutions assigned to you by your instructor. Add a magnetic stirring bar and begin stirring. Use a buret to add 50 mL of 0.7M aqueous ammonia (NH_3) drop by drop to the solution. A black precipitate of magnetite should form as you slowly add the ammonia solution over a period of about 5 minutes.

2. Turn off the stirrer and quickly use a strong magnet to slide the stir bar up the walls of the beaker. Remove the stir bar with gloved hands **before** it touches the magnet.

3. Let the magnetite settle for a few minutes, then decant and discard the clear liquid, making sure to save as much of the solid as possible. Transfer the solid to a plastic weighing boat. It may be necessary to rinse the beaker with water from a wash bottle.

4. Use a strong magnet to hold the magnetite to the bottom of the weighing boat and once again decant as much of the clear liquid as possible. Repeat this step two more times.

5. Add 2.0 mL of 25% tetramethylammonium hydroxide and stir with a glass stir rod to suspend the solid in the liquid. Using the strong magnet to hold the ferrofluid to the bottom of the weighing boat, pour off and discard the liquid. Move the magnet around and pour off the remaining liquid.

6. Place a cow magnet or other very strong magnet under the remaining fluid and record your observations relative to its spiking behavior. If the fluid does not spike readily, then add **one drop** of water and try again. Share your ferrofluid results with the rest of the class and be sure to observe the spiking behavior of the ferrofluids produced by your classmates.

DATA TABLE I

Group	1	2	3	4	5	6	7
mL 1 M $FeCl_3$	1	2	3	4	5	6	7
mL 2M $FeCl_2$	1	1	1	1	1	1	1
"spiking" (Y or N)							

ANALYZING THE DATA

1. Determine the mole ratio of $FeCl_3$ to $FeCl_2$ and enter in the table below. Show your work!

GROUP	1	2	3	4	5	6	7
RATIO							

2. Which of the ratios from the table above corresponds to the ferrofluids that produced the best spiking behavior?

3. Use your answer to question (2) above to balance the equation for the synthesic reaction below.

___$FeCl_3$ +___ $FeCl_2$ +___NH_3 +___H_2O →___Fe_3O_4 +___ NH_4Cl