EXPERIMENT 1
Notes for the Instructor

PURPOSE

To synthesize magnetite nanoparticles in aqueous solution using a variety of $FeCl_2$ to $FeCl_3$ mole ratios. Once prepared, the nanoscale particles are coated with the surfactant tetramethylammonium hydroxide to create a ferrofluid whose appearance is altered reversibly by the presence of a strong magnetic field. The "spikes" that appear in the presence of the magnet can be used as a rough measure of the success of the synthesis and as a means for estimating the stoichiometry of the reaction.

METHOD

The students follow a general procedure for the production of aqueous- based ferrofluids with the exception that they will use various ferrous-to-ferric mole ratios in the synthesis. Students will then decide which of the resulting fluids produces spikes similar to those they observed in Investigation 1 and thereby estimate the correct stoichiometric ratio for the reactants.

A pre-lab discussion could include a discussion of nanoparticles and surfactants. For the surfactant employed in this experiment, tetramethylammonium hydroxide, a reasonable expectation is that the hydroxide anions could bind to iron atoms at the surface of the magnetic nanoparticles and tetramethylmmonium cations could form a sheath around the negatively-charged particles through electrostatic attraction. These shesths of positive charge can prevent the particles from agglomerating. The magnetic properties of the magnetite particles are also noteworthy but the level of discussion needs to reflect student familiarity with electron configurations. If the main focus of the experiment is on stoichiometry, discussions of electron spins can be deferred and revisited later in the course. See Appendix A or the background information for this module for a more complete discussion of this topic.

MATERIALS

Magnetic stirrers and stir bars

Cow magnets (0.5 in. x 3 in.), available from farm supply stores or from Edmund Scientific, Barrington, NJ.

Strong magnet (neodymium-iron-boron), available from the physics department or Edmund Scientific. Computer hard drive magnets will also work.

2.0 M $FeCl_2$ in 2 M HCl

1.0 M $FeCl_3$ in 2 M HCl

0.7 M NH_3

25 % aqueous tetramethylammonium hydroxide solution, available from Aldich, Milwaukee, WI.

ANSWERS TO 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	**0.5:1**	**1:1**	**1.5:1**	**2:1**	**2.5:1**	**3:1**	**3.5:1**

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

   Students should find that stoichiometric ratios from 1.5:1 to 2.2:1 yield spikes, but outside these parameters little or no spiking occurs. The *best* results, however, are observed with a 2:1 ratio.

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

   _2_$FeCl_3$ + **_1_**$FeCl_2$ + **_8_**NH_3 + **_4_**H_2O → **_1_**Fe_3O_4 + **_8_** NH_4Cl