Laboratory Objectives: The objective of this laboratory are to experimentally evaluate the solubility of aluminum following the precipitation of an aluminum hydroxide solid and to examine the effect of a complexing ligand on aluminum solubility. Experimental results will be compared with predictions based on chemical equilibrium calculations. Aluminum hydroxide will be precipitated by addition of strong base to a soluble aluminum salt solution, and the resulting suspension will be adjusted to several pH values. Students will be divided into two groups; one group will examine aluminum solubility in the absence of the complexing ligand and the other will examine the solubility when the ligand is present. Laboratory data collected by each group will be distributed to all students for independent interpretation.

Equipment Needed
- pH meter and combination pH electrode
- magnetic stirrer with magnetic stir bars (6)
- glass beakers (8)
- syringes (2) and 0.2 μm syringe filters (6)
- graduated cylinder
- pipettors and pipette tips
- plastic test tubes

Reagents
The following reagents will have been prepared before the laboratory session by the instructor.
- 0.1M Al₂(SO₄)₃ solution
- 0.1M Citric Acid Solution
- 1M NaOH
- 1M HNO₃
- concentrated HNO₃
- 1% HNO₃
- Aluminum Calibration Standards

Procedures
1. Prepare six 50 mL volumes of 0.01 M Al₂(SO₄)₃ (group 1) or six 50 mL volumes of 0.01 M Al₂(SO₄)₃ plus 0.002 M citric acid. Add deionized water to bring the total volume to 50 mL.
2. Measure and record the pH in at least two of these initial solutions.
3. Add 1 M NaOH to the reactors to bring each one to a different final pH. Record the amount of NaOH added. The target pH values are 4, 5, 6, 7, 8, and 9. Try to get within 0.3 pH units of the target value. If you overshoot the pH, you can use the 1 M HNO₃ to reduce the pH. After the target pH is reached, allow the solution or suspension to sit for at least 10 minutes.
4. Collect a dissolved sample by pulling 10 mL from each beaker with a syringe and filtering the solution using the syringe filter. Add 0.1 mL of concentrated acid to the filtered solution to preserve it before analysis.
5. Qualitatively observe the time that it takes for each suspension to settle.
6. Take your samples to the analytical lab (Urbauer Room 13) to have them run on the inductively coupled plasma mass spectrometer (ICP-MS). If you think that the concentration of aluminum in a sample will be higher than 500 ppb (500 μg/L), then dilute your sample with 1% HNO₃.
Data Preparation
The dissolved aluminum in each sample should be calculated by using a calibration curve developed with the ICP-MS calibration standards (the results of the calibration standards will be provided to you). Ultimately you will want to report these results in units of moles per liter.

Data Analysis
The following analyses should be performed and incorporated in the Results and/or Discussion sections of the laboratory report in an organized and appropriate manner. For the items below, complete them for both sets of experiments (i.e., with and without the presence of citrate).

(i) Calculate the initial pH of the solutions. How does the calculated initial pH compare with the initial pH measured for each solution? If there is a deviation, how might this be explained?
(ii) For each target pH, calculate the NaOH needed to reach that pH. How do these values compare with those observed in your experiments?
(iii) Calculate the dissolved aluminum concentrations expected to be in equilibrium with Al(OH)$_3$(s). Compare the measured concentrations with those that you calculate. Show calculations and results on a logC-pH plot. Comment on whether or not the systems are at equilibrium.
(iv) Calculate the solubility of aluminum in equilibrium with another aluminum oxide, hydroxide, or oxyhydroxide solid. Compare those values with the equilibrium solubility calculated for Al(OH)$_3$(s) and also with the values you measured.
(v) Qualitatively discuss the effect of pH on the settling velocity of the precipitate, and suggest possible explanations for any effects that you observe.

Report Preparation
Prepare your report according to the specifications in the document “Preparation of Laboratory Reports” by David A. Dzombak, October 1995.

Reports will be evaluated based on:
1. Abstract (5 points)
2. Introduction (10 points)
3. Background/Theory (10 points)
4. Experimental Methods (10 points)
5. Results (15 points: prose (5 points) and graphical/tabular (10 points))
6. Discussion of Results (15 points)
7. Summary and Conclusions (10 points)
8. Table of Contents; References (5 points)
9. Appendices (presentation of raw data, data reduction, other supporting material) (10 points)
10. Overall organization and clarity of report (10 points)
Pre-lab Assignment:

1. Calculate the volumes of 0.1M Al$_2$(SO$_4$)$_3$ and 0.1M citric acid solution needed to prepare the 50 mL volumes of 0.01 M Al$_2$(SO$_4$)$_3$ or of 0.01 M Al$_2$(SO$_4$)$_3$ plus 0.002 M citric acid.

2. Calculate the initial pH of each solution.

3. Calculate the volume of 1 M NaOH needed to raise the pH of each solution to 4, 5, 6, 7, 8, and 9. Assume that Al(OH)$_3$(s) is the only solid that will form.

4. Calculate the dissolved aluminum concentrations expected to be in equilibrium with Al(OH)$_3$(s) for each solution.