1. Aluminum is determined gravimetrically by precipitating $Al(OH)_3$ and isolating Al_2O_3. A sample containing approximately 0.1 grams of Al is dissolved in 200 mL of H_2O, and 5 grams of NH_4Cl and a few drops of methyl red indicator are added (methyl red is red at pH levels below 4 and yellow at pH levels above 6). The solution is heated to boiling and 1:1 NH_3 is added dropwise till the indicator turns yellow, precipitating $Al(OH)_3$. The precipitate is held at the solution's boiling point for several minutes before filtering and rinsing with a hot solution of 2% w/v NH_4NO_3. The precipitate is then ignited at 1000–1100 ºC, forming Al_2O_3.
2. Mixing solutions of $Ba(SCN)_2$ and $MgSO_4$ produces a precipitate of $BaSO_4$. Shown below are the descriptions and results for three experiments using different concentrations of $Ba(SCN)_2$ and $MgSO_4$. Cite two ways in which this procedure encourages the formation of larger particles of precipitate. The ignition step must be carried out carefully to ensure the quantitative conversion of $Al(OH)_3$ to Al_2O_3. What is the effect of an incomplete conversion on the %w/w Al? What is the purpose of adding NH_4Cl and methyl red indicator? An alternative procedure involves isolating and weighing the precipitate as the 8-hydroxyquinolate, $Al(C_9H_6ON)_3$. Why might this be a more advantageous form of Al for a gravimetric analysis? Are there any disadvantages?
3. Calcium is determined gravimetrically by precipitating $CaC_2O_4 \bullet H_2O$ and isolating $CaCO_3$. After dissolving a sample in 10 mL of water and 15 mL of 6 M HCl, the resulting solution is heated to boiling and a warm solution of excess ammonium oxalate is added. The solution is maintained at 80 ºC and 6 M NH_3 is added dropwise, with stirring, until the solution is faintly alkaline. The resulting precipitate and solution are removed from the heat and allowed to stand for at least one hour. After testing the solution for completeness of precipitation, the sample is filtered, rinsed with 0.1% w/v ammonium

oxalate, and dried at 100–120 ºC for 1 hour. The precipitate is transferred to a muffle furnace where it is converted to $CaCO_3$ by drying at 500 ± 25 ºC until constant weight.

a. Why is the precipitate of $CaC_2O_4 \bullet H_2O$ converted to $CaCO_3$?

b. In the final step, if the sample is heated at too high of a temperature some $CaCO_3$ may be converted to CaO. What effect would this have on the reported %w/w Ca?

c. Why is the precipitant, $(NH_4)_2C_2O_4$, added to a hot, acidic solution instead of a cold, alkaline solution?

4. Iron is determined gravimetrically by precipitating as $Fe(OH)_3$ and igniting to Fe_2O_3. After dissolving a sample in 50 mL of H_2O and 10 mL of 6 M HCl, any Fe^{2+} is converted Fe^{3+} by oxidizing with 1–2 mL of concentrated HNO_3. The sample is heated to remove the oxides of nitrogen and the solution is diluted to 200 mL. After bringing the solution to a boil, $Fe(OH)_3$ is precipitated by slowly adding 1:1 NH_3 until the odor of NH_3 is detected. The solution is boiled for an additional minute and the precipitate is allowed to settler. The precipitate is then filtered and rinsed with several portions of hot 1% w/v NH_4NO_3 until no Cl^- is found in the wash water. Finally, the precipitate is ignited to constant weight at 500–550 ºC and weighed as Fe_2O_3.

a. If ignition is not carried out under oxidizing conditions (plenty of O_2 present), the final product may contain Fe_3O_4. What effect would this have on the reported %w/w Fe?

b. The precipitate is washed with a dilute solution of NH_4NO_3. Why is NH_4NO_3 added to the wash water?

c. Why does the procedure call for adding NH_3 until the odor of ammonia is detected?

d. Describe how you might test the filtrate for Cl^-.

5. A sample of an impure iron ore is approximately 55% w/w Fe. The amount of Fe in the sample is to be determined gravimetrically by isolating it as Fe_2O_3. What mass of sample do you need to ensure that you isolate at least 1 g of Fe_2O_3?
6. The concentration of arsenic in an insecticide is determined gravimetrically by precipitating $MgNH_4AsO_4$ and isolating $Mg_2As_2O_7$. Determine the %w/w As_2O_3 in a 1.627-g sample of insecticide if it yields 106.5 mg of $Mg_2As_2O_7$.

7. After preparing a sample of alum, $K_2SO_4 \bullet Al_2(SO_4)_3 \bullet 24H_2O$, a student determined its purity by dissolving a 1.2391-g sample and precipitating the aluminum as $Al(OH)_3$. After filtering, rinsing, and igniting, 0.1357 g of Al_2O_3 is obtained. What is the purity of the alum preparation?