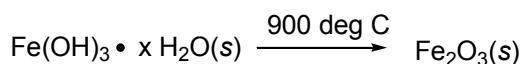
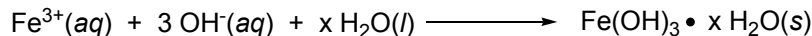


## Experiment 1: Gravimetric Determination of Iron as Fe<sub>2</sub>O<sub>3</sub>

(Adapted from Harris's *Quantitative Chemical Analysis*)

### Experimental Work in Olin-Rice 380 (and 378) on February 2 and 9 Notebook Due on February 20 by 5 p.m. (20% per day penalty if late)

As we discussed during the first week of classes, a sample containing iron can be analyzed by precipitation of the hydrated hydroxide from basic solution, followed by ignition to produce Fe<sub>2</sub>O<sub>3</sub>:

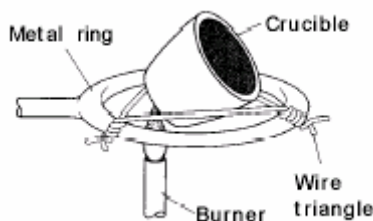


You will use this gravimetric technique to determine the weight percent of Fe in an iron ore sample. You will make three replicate measurements, which will allow you to determine both an average and a 95% confidence interval for your data.

### PROCEDURE

**General Comments:** (a) This is a two-week experiment. (b) You and your lab partner(s) should be strategic about dividing up the tasks for this experiment. Specifically, you should be doing Steps 1 and 2 at the same time. (c) Never insert a pipet into any common reagent bottle. Instead, pour out some for yourself in a beaker. (d) We will be spread out between Olin-Rice 378 and Olin-Rice 380. Each lab team should have two gas jets to speed up the crucible heating.

1. Bring three porcelain crucibles and caps to constant mass by heating over a Bunsen burner (Figure 1). (Each group will have two gas jets, so you should be heating two of your crucibles at the same time.) Increase the amount of air being mixed with the fuel until you see an inner light blue cone in the flame—this is the hottest part of the flame. Adjust the height of the metal ring to expose the crucible to that light blue cone.



**Figure 1.** Positioning a crucible above a burner. Note that the tilt is important! You want to maximize the exposure of the crucible to the flame.

The crucibles should be heated to redness for 15 min. (Be sure that all oxidizable substances on the entire surface of each crucible have burned off.) Then cool each crucible for 30 min in a desiccator (to minimize the adsorption of water vapor onto the crucible) and weigh each crucible on an analytical balance. Avoid touching the crucibles

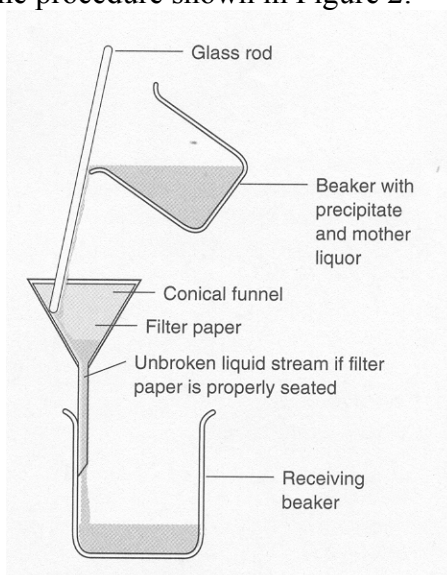
with your bare fingers; the weight of your fingerprints will register on the balance! Then repeat the above process: 15 min of heating to redness, 30 min of cooling in a desiccator, and weighing on an analytical balance. Your two weighings should agree to within 1 mg. If not, you should repeat the process once more. (Usually the last weight is the most accurate.) When you are done weighing your crucibles, store them in a desiccator and put them away in your cabinet for use next week.

2. Meanwhile, accurately weigh three samples from one of the unknown vials. Each sample should be around ~0.3 g. Put each sample in a 400-mL beaker, and fit all three beakers onto a large hot plate in a fume hood. Wearing acid gloves, dissolve each sample in 10 mL of concentrated (~12 M) HCl. Heat gently with stirring. (Keep the rubber policemen out of the solution—we don't want bits of rubber in our samples!) More acid may be necessary to completely dissolve the samples. Make sure that you do not boil off all the water! It is easy to completely cook your samples, which means you will have to start over. If there are insoluble impurities, filter through qualitative filter paper and wash the filter very well with distilled water. (This will usually not be necessary.)

3. Add 5 mL of 6 M HNO<sub>3</sub> to each sample, and boil for a few minutes to ensure that all iron is oxidized to Fe(III). Again, be careful not to boil away all the water!

4. Dilute the sample to 200 mL with distilled water and add 3 M ammonia with constant stirring until the solution is slightly basic (as determined with litmus paper or pH indicator paper). Digest the precipitate by boiling for 5 min and allow the precipitate to settle. (Alternatively, if the sample never comes to a boil, it is sufficient for the solution to have been heated vigorously for 25 min.)

5. Set up a filter rack and fit three long-necked funnels with coarse, ashless filter paper (Whatman 41, 110-mm diameter). Wet paper so that it will stick to the funnel. For each sample, decant the supernatant liquid (that is, the liquid above the precipitate) through one of the filters. Follow the procedure shown in Figure 2:



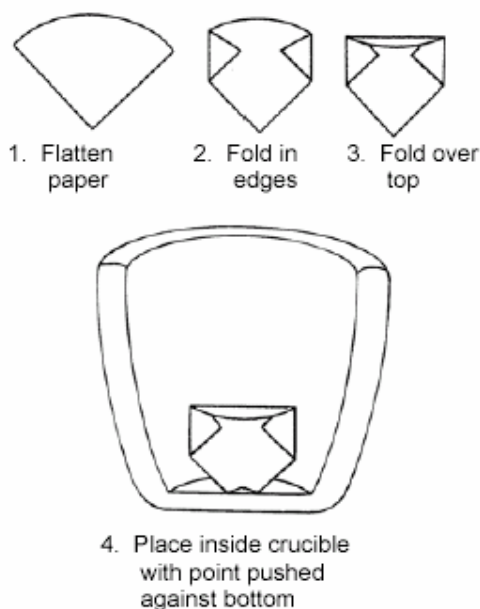
**Figure 2:** Filtering a precipitate. See Section 2-7 in your textbook for more details.

Do not pour liquid higher than 1 cm from the top of the funnel. Transfer any remaining solid from the 400-mL beaker to the filter paper with the aid of a rubber policeman and with hot 1%  $\text{NH}_4\text{NO}_3$  (you will need to heat your own). Wash the precipitate with more hot 1%  $\text{NH}_4\text{NO}_3$ . Test for  $\text{Cl}^-$  in each precipitate by transferring a small volume of filtrate from each receiving beaker to a well (on a wellplate), and then adding a few drops of 0.1 M  $\text{AgNO}_3$ . If you see a milky white color, indicating the formation of  $\text{AgCl}(s)$ , you still have a significant amount of  $\text{Cl}^-$  in your precipitate. Clean out the receiving beaker for that precipitate, and wash the precipitate with more hot 1%  $\text{NH}_4\text{NO}_3$ . Repeat until the white color is non-existent or faint.

6. Cover each of the funnels with a ribbed watch glass. This will both protect each precipitate from dust and allow it to dry during the week. If you are done with your crucible weighings (Step 1), you are done for the week!

**WASTE DISPOSAL FOR WEEK 1:** The well plate solutions, which contain  $\text{Ag}^+$ , must go into waste bottles. Everything else can go down the drain with lots of water.

7. (**Week 2**) Don't be surprised at how much volume has been lost from your hydrated iron samples! Carefully lift the paper out of the funnel, fold it (Figure 3), and transfer it to one of your accurately weighed crucibles. Fold up the filter papers as much as possible. Do not let the paper stick out over the top edge of the crucible.



**Figure 3.** Folding filter paper and placing it inside a crucible for ignition. Continue folding paper so entire package fits at the bottom of the crucible. Be careful not to puncture the paper.

8. Dry each crucible cautiously with a small flame, as shown in Figure 1. The flame should be directed at the top of the container. Avoid spattering. After it is dry, *char* (or blacken) the filter paper by increasing the flame temperature. (However, take the flame away if the paper starts to inflame.) Once the paper is charred, you can be even more aggressive and aim the flame directly at the paper. Any carbon left on the crucible

should be burned away by directing the burner flame at it. To make your job easier, try to avoid spreading out charred residue over a lot of the inner surface of the crucible. Use tongs to manipulate the crucible. Finally, *ignite* the product for 15 min with the full heat of the burner. Again, to maximize efficiency, have two burners going simultaneously.

9. Cool each crucible briefly in air and then in a desiccator for 30 min. Weigh the crucible, reignite, and bring to constant mass (within 1 mg) with repeated heatings. However, like last week, you are required to do a maximum of (only) three weighings for each crucible.

**WASTE DISPOSAL FOR WEEK 2:** All iron waste can go into the trash.

**DATA ANALYSIS:** Calculate the weight percent of iron in each sample, and the average, the standard deviation, and the 95% confidence interval for the set of three weight percent values. You should use the last weight obtained for each crucible in your calculations. Note that the uncertainty will be determined solely by the variation in the weight percent of iron in your three trials--do not propagate uncertainties in the weights of the crucibles. You will also perform a Case 1 *t*-test, comparing your experimental average to the accepted value I will e-mail you after the February 9 lab session. Discuss the possible sources of both systematic and random error in the experiment, and judge what the dominant sources may be. Identify the sign of each systematic error, and try to estimate its magnitude. Note that even if your Case 1 *t*-test does not indicate the presence of systematic error, you should still discuss what the likely sources of systematic error would be.

Remember that your lab notebooks are due on (or before) February 20 at 5 p.m.