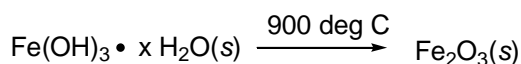
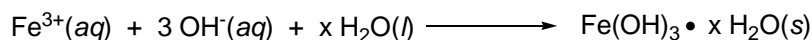


Experiment 1: Gravimetric Determination of Iron as Fe₂O₃

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

Experimental Work in Olin-Rice 380 (and 378) on February 1 and 8 Notebook Due on February 19 by 4:00 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 Fe₂O₃:



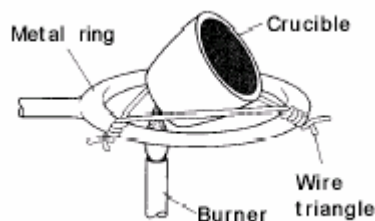
You will use this gravimetric technique to determine the weight percent of Fe in an iron ore sample. You will make three replicate measurements, which will allow you to determine both an average and a 95% confidence interval for your data.

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. You should start in on step 2 while waiting for parts of step 1.

1. Bring three porcelain crucibles and lids 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 a lid should be in place as well. This setup will be demonstrated at the start of the first week of this experiment.



Each crucible and lid pair should be heated to redness for 15 min. That is, you should heat until you see all of the ceramic in the crucible glowing red, and then wait for 15 min past that point. It will take a lot longer to heat to redness if the crucible is not being exposed to the light blue part of the flame. (We have brand new alumina crucibles that are rated to withstand temperatures up to 1750°C, which is significant hotter than what we can generate with our Bunsen burners.) Then cool each crucible and lid for 30 min in a desiccator (to minimize the adsorption of water

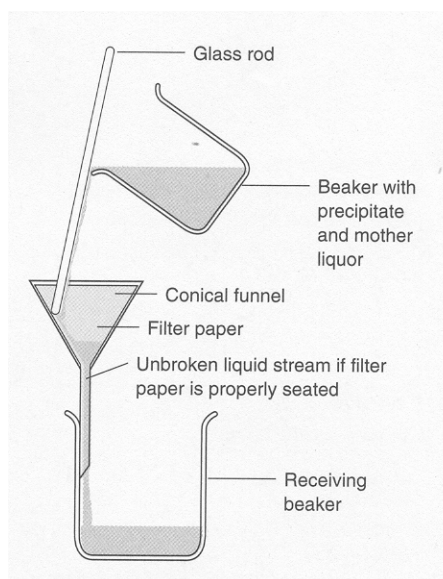
vapor onto the crucible and lid) and weigh each crucible and lid *independently* on an analytical balance. Avoid touching these with your bare fingers; the weight of your fingerprints will register on the balance! Next, repeat the above process: 15 min of heating to redness, 30 min of cooling in a desiccator, and weighing on an analytical balance. Your two weighings should agree to within 1.0 mg for each piece. If not, you should repeat the process once more. (Usually the last weight is the most accurate.) When you are done weighing your crucibles and lids, store them in a desiccator and put them away in your cabinet for use next week. Make sure the lid of the desiccator seals tightly

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 paper very well with distilled water. (This will usually not be necessary.)

3. Add 5 mL of 6 M HNO₃ 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.)

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



5. Set up a filter rack and fit three long-necked funnels with coarse, ashless filter paper (Whatman 41, 110-mm diameter). Make sure you choose the ashless paper, since qualitative

filter paper will also be around! 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 above. 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% NH_4NO_3 (you will need to heat your own). Wash the precipitate with more hot 1% NH_4NO_3 . Test for 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 AgNO_3 . If you see a milky white color, indicating the formation of $\text{AgCl}(s)$, you still have a significant amount of Cl^- in your precipitate. Rinse out the receiving beaker for that precipitate with DI water, and wash the precipitate with more hot 1% NH_4NO_3 . Repeat until the white color of AgCl seen in the spot plate test becomes 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 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! Take a crucible and lid out of your dessicator, and weight them yet again. Carefully lift the paper out of the funnel containing one of your precipitates, fold it (as shown in Figure 3 below), and transfer it to the crucible. Fold up the filter papers as much as possible. We are about to burn away the filter paper, and the tighter you can fold your samples, the easier it will be to remove the paper cleanly. Do not let the paper stick out over the top edge of the crucible, or you risk losing some of your sample. Repeat this process for each of your precipitate samples.

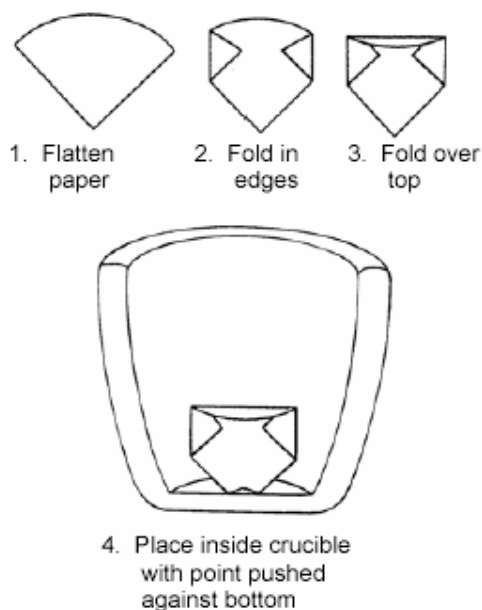


Figure 3. Folding filter paper and placing it inside a crucible for ignition. Continue folding paper so that the entire sample fits at the bottom of the crucible. However, be careful not to puncture the paper.

8. Set up the crucibles and lids as you did last week. Be sure that the lids are tilted slightly, so that $\text{H}_2\text{O}_{(g)}$ and $\text{CO}_{2(g)}$ can leave the crucibles, and $\text{O}_{2(g)}$ can enter the crucibles. Adjust your Bunsen burner to supply a cool flame—that is, one without a light blue inner cone. Dry each of your samples by heating the crucibles with this cool flame. Minimize spattering. (The job of the lid is to contain whatever spattering does happen.)

9. After each sample is dry, increase the flame temperature, and heat the crucibles until the filter paper is blackened. Take the flame away if the paper catches fire. Be sure air can get into the crucible, or the heat you are supplying will end up turning the carbon in the filter paper to graphite (which will stick to your analyte) instead of carbon dioxide. Once the paper is charred, be more aggressive and aim the flame directly at the paper. Any carbon left on the crucible or the underside of the lid 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 and lid. Finally, expose your samples to the inner blue cone of the flames. Like last week, we now seek to heat the crucibles to redness for 15 min with the full heat of the burner. Again, to maximize efficiency, have two burners going simultaneously.

10. Cool each crucible and lid briefly in air and then in a desiccator for 30 min. Weigh the crucible and lid, heat the crucible to redness, and bring to constant mass (within 1.0 mg) with repeated heatings. However, like last week, you are required to do a maximum of (only) three weighings for each crucible and lid.

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 8 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 19 at 4:00 p.m.