

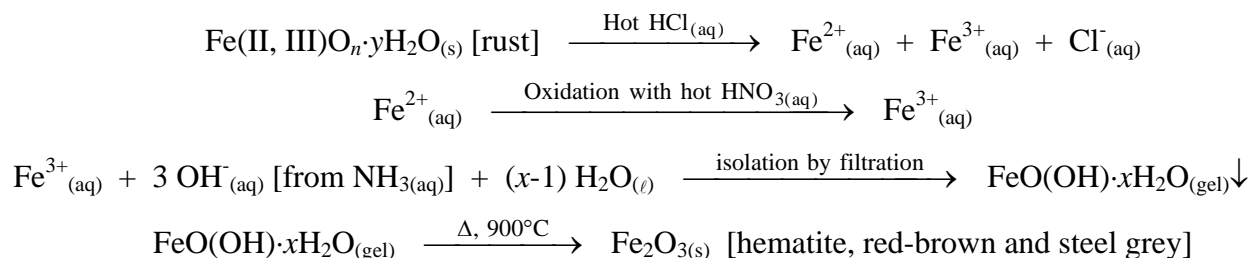
## Experiment 1: Gravimetric Determination of Iron as Fe<sub>2</sub>O<sub>3</sub>

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

**Experimental Work in Olin-Rice 380 (and 378) on January 29, February 5 and 12  
Notebook Due on February 23 by 4:00 p.m. (20% per day penalty if late)**

### INTRODUCTION

As we will discuss during the first week of classes, a sample containing various oxides of iron can be analyzed by dissolution, oxidation to a single oxidation state, precipitation of the hydrated hydroxide from basic solution, and finally 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 at least three replicate measurements, which will allow you to determine both an average and a 95% confidence interval for your data.

### SAFETY ISSUES AND LOGISTICS

- **This lab INVOLVES CHEMICALS THAT POSE A SUBSTANTIAL RISK IF NOT HANDLED PROPERLY. Read and heed safety instructions here and in the experimental procedure**
- **Concentrated hydrochloric acid is highly corrosive, and gives off toxic, corrosive HCl(g) fumes, particularly when heated, as you will do in this lab, *in a fume hood*.**
- **Nitric acid (HNO<sub>3</sub>) is a powerful acid and oxidizing agent, particularly when hot (as it will be in this experiment!)**

### WASTE

- The only chemical involved in this experiment that is not compatible with the sewer system by the time it needs to be disposed of is the silver used in week 2. Please carefully follow the instructions for its disposal. If you end up with a significant amount of un-neutralized acid or base that needs to be disposed of, please consult with your lab instructor for neutralization instructions.

### EXPERIMENTAL PROCEDURE

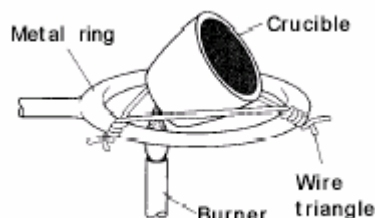
This is a three week experiment; in the first week, you will heat and dry your crucibles while checking into your lab drawers. In the second week, you will complete the preparation of your crucibles and begin the preparation of your samples. In the third and final week, you will transfer your samples into your crucibles and burn away anything volatile, in preparation for your final mass determinations, which are most efficiently done on a few minutes of your own time, once the samples have cooled.

We will be spreading out between Olin-Rice 378 and 380 in order to have enough gas jets for all the heating we are going to do. We'll also prop open the doors, because the lab will get *hot!*

### Week 1 – Crucible Preparation and Lab Drawer Check-In

1. Extract the dessicator from your lab drawer and open it by sliding the lid off. Your lid might be stuck from a year of non-use, so ask for help if it doesn't open easily. Wearing gloves, remove the crucibles inside and put them on a clean paper towel. If the indicating Drierite™ inside the dessicator is pink rather than blue, ask your instructor or TA for assistance in replacing it. If any of the crucibles look to have cracks that threaten their integrity, please ask for a consultation.
2. Place four sound crucibles in one dessicator, in such a manner that their positions identify which one is which. (Most likely, numbers have already been written on the insert inside the dessicator; if not, that is the best way to keep them straight. Don't write on the crucibles!)
3. Bring this dessicator into the weighing room, and put it next to an unoccupied analytical balance. Weigh your four crucibles, noting whether each is made of porcelain or alumina. This will give you a baseline value, to determine if heating is removing any fingerprint oils and/or absorbed water from the crucible. Put the crucibles back in the dessicator and return to your bench.
4. Heat each crucible over a Meker-type (mongo-huge) 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 a dancing light blue set of small flames right at the burner head surface. Adjust the height of the metal ring so that the crucible is positioned just above that bright blue layer. (CAUTION: Do this before the ring gets too hot to handle!)

**Figure 1.** Positioning a crucible above a burner. This setup will be demonstrated at the start of the first week of this experiment. You will be using a much bigger burner, with a larger flame area...which works much better! Putting the crucible on its side reduced the thermal strain it has to endure.



5. Each crucible should be kept at orange heat for 5 minutes. You should heat until you see all three of the ceramic supports on the clay triangle glowing orange, and then wait for 5 minutes past that point. (It's safest for your crucibles to heat them gradually, bringing the flame in and out from under the crucible every few seconds when you first start heating.) When time is up, turn OFF the Bunsen burner and let the entire setup sit for 30 minutes. **Everything is super-hot, and dangerous to handle, so don't touch anything, just turn off the heat and let everything cool.**
6. While waiting for your first two crucibles to cool, check into your lab drawer.
7. Once the first pair of crucibles has cooled for 30 minutes in the air, transfer this pair of crucibles into your dessicator. Avoid touching the crucibles with your bare fingers, as the

weight of your fingerprints will register on the balance! Extract the other two crucibles, and place them on the metal rings over the burners.

- Repeat the heating procedure with the remaining two crucibles, again allowing them to cool in the air for at least 30 minutes before placing them into the dessicator. While waiting, clean any glassware in your drawer that looks as though it might need it. Use water, but not soap!
- Close the dessicator, making sure the lid of the desiccator seals tightly, and that your indicating Drierite crystals are blue, not pink. That's it for today!

## Week 2 – Sample and Crucible Preparation

- Accurately weigh four samples from one of the unknown vials. Each sample should be around ~0.3 g. Put each sample in a *clean, dry* 400-mL beaker, and fit all four beakers onto a large hotplate in a fume hood. Wearing acid gloves, dissolve each sample in 20 mL of concentrated (~12 M) HCl. Heat gently with stirring. (Keep the rubber policeman out of the solution—we don't want bits of oxidized 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. In the unlikely event there are any insoluble impurities in your sample, you will need to filter through qualitative filter paper and wash the filter paper very well with distilled water. Ask your lab preceptor or instructor for help with this step, if you think it may be necessary.
- 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!
- Dilute the sample to 200 mL with distilled water and add 3 M ammonia with constant stirring until the solution is slightly basic (as indicated by a pH probe). Be sure to rinse the probe back into the beaker before removing it, so as to not lose any iron! Digest the precipitate (flocculate it!) 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.)
- Set up a filter rack and fit four long-necked funnels with coarse, ashless filter paper (Whatman 41, 110-mm diameter) into them. Wet the filter paper so that it sticks to the funnel. For each sample, stir and pour the solution into the funnel, trying to transfer as much suspended solid as possible with each pouring. The less solid you leave behind in the beaker the better, but don't spill, splash, or splatter any of the brown goo out of the funnel! 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%<sub>mass</sub> NH<sub>4</sub>NO<sub>3</sub> (you will need to heat your own). Wash the precipitate with more hot 1%<sub>mass</sub> NH<sub>4</sub>NO<sub>3</sub>. Test for Cl<sup>-</sup> 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<sub>3</sub>. If you see a milky white color, indicating the formation of AgCl(s), you still have a significant amount of Cl<sup>-</sup> in your precipitate. Wash the precipitate with more hot 1%<sub>mass</sub> NH<sub>4</sub>NO<sub>3</sub>. Repeat until the white color of AgCl seen in the spot plate test becomes non-existent or faint. While waiting for the filtration, do the next step.

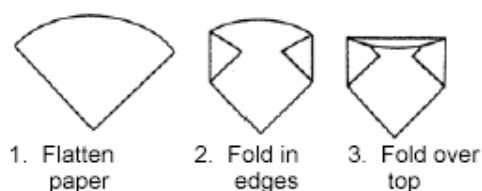
14. Extract your crucibles from your dessicator and weigh them. If the mass of any of the crucibles has changed by more than 0.0001 g since the last weighing, you will need to bring that crucible through the heating and cooling process again. (Whether you need to do this depends on what was done with the crucible before it came into your possession, so don't feel you made a mistake if the mass changed a lot after the first heating. However, if done right, it only takes one heating to completely bake out a crucible! So the second time you heat a crucible *should* be the last.) Be certain that each crucible cools at least 30 minutes in the air before putting it into the dessicator. There's no point in re-weighing the crucibles yet again until they have *thoroughly* cooled. You can do this on your own, or wait until the start of week 3 to do it.
15. Once each of the filtrates emerge free of chloride ions, 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 and re-heatings (described in the step immediately above), you are done for the week!

### WASTE DISPOSAL FOR WEEK 2

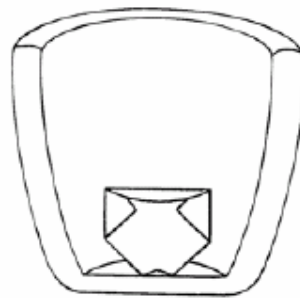
- The well plate solutions, which contain  $\text{Ag}^+$ , **cannot** go down the drain. Please just leave your well plate at your lab station, with the silver-containing solutions in it.
- Everything else from this lab can be poured down the drain.

**Week 3 – Sample Ignition**

16. Don't be surprised at how much volume has been lost from your hydrated iron samples! Take a crucible out of your desiccator, and weigh it yet again. If for some reason it does not agree with the previous weighing to within  $\pm 0.0001$  grams, please consult your instructor. Carefully lift the paper out of the funnel containing one of your precipitates, using a spatula to free it from the funnel. Fold it (as shown in Figure 2 below), and transfer it to the crucible. Fold up the filter papers as much as possible, but above all else, don't lose any of the solid from within! 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, but don't over-do it at the expense of your sample's integrity! 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 2.** 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.



4. Place inside crucible with point pushed against bottom

17. Set up the crucibles over the burners as you did when heating them to constant mass. Do this without the burner lit – you want to heat things up quickly once you are actually ready.
18. Light the burner and put it under the crucible; in this case, don't bring it in and out from under the crucible, just put it underneath and leave it there, at full heat. With the flame so hot, the filter paper should quickly catch fire: this is actually desirable, as it minimizes smoke (which can carry away some iron) and also prevents the formation of graphite due to incomplete combustion of the filter paper. [Be sure air can readily 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.] Heat the crucibles to redness for 15 min, using the full heat of the burner, to ensure complete ignition of the iron oxide. Depending on the conditions under which the ignition takes place, the resulting hematite may be dark red, steel grey, or (most likely) a combination of these two. Again, to maximize efficiency, have two burners going simultaneously.
19. Cool each crucible at least 30 minutes in the air before transferring it to the desiccator. Once all four samples have been ignited and the crucibles cooled, you can seal up your

dessicator and call it a day. (The crucibles need to cool completely before their mass can be accurately measured.)

20. You will need to come back sometime later to weigh your crucibles containing the  $\text{Fe}_2\text{O}_3$ .

### WASTE DISPOSAL FOR WEEK 3

- All iron waste can go into the trash.

### DATA ANALYSIS:

1. Work through the analysis of the %<sub>mass</sub> Fe in one of your samples in detail, propagating error quantitatively through the calculations as described in Chapter 3 of Harris. You will first calculate the mass of the empty crucible, and the quantitative uncertainty in that value – either based on the reproducibility of the measurement, or (if you did not get three seemingly randomly-varying results for the mass) on the uncertainty in the measuring devices used to obtain it. You will then need to calculate the mass of  $\text{Fe}_2\text{O}_3$  obtained in that crucible, along with the uncertainty in that value. Finally, you'll calculate the %<sub>mass</sub> Fe for that sample, based on the mass of the sample and the molar masses of the species involved, each with their associated uncertainties. Please use the periodic table in Harris in determining your molar masses and their uncertainties. Propagate error quantitatively, in detail, throughout these calculations. It's not that hard to do! You may assume this to reflect the uncertainty inherent in each of your individual results.
2. Calculate the weight percent of iron in each sample, and the average, the standard deviation, and the 95% confidence interval for your set of four weight percent values. (If you only ended up with three, that's OK! We did four to cover your bum in the case one of your trials should go south.) You should use the last weight obtained for each crucible in your calculations.
3. Perform a Case 1  $t$ -test, comparing your experimental average to the accepted value I will e-mail you after the February 12 lab session.
4. 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. It will be valuable to talk with others about the trends in their data!

Your data analysis should be presented with a clear and complete sample calculation in your lab notebook. You are free to use a spreadsheet, but a hand-written sample calculation clearly demonstrating how you did the calculations is imperative, and will be graded.

**Remember that your lab notebooks are due on (or before) February 23 at 4:00 p.m.**