# APPENDIX: GUIDELINES FOR THE IRON SALT REPORT AND CALCULATIONS

This scientific report should be a concise and clear presentation of **YOUR** observations, data, calculations, and conclusions for the experiment. Your goal in a report should be to inform the reader of what you wanted to do (introduction), of how you did it (experimental), of what you did (results), and of what the result mean (discussion) but for this report we will only focus on the results and the discussion of the results. If the reader is to be properly informed, the report should be readable, both grammatically and legibly. An appendix with copies of notebook pages and a grading matrix will also be part of this report.

Reports may have different forms depending on the information being conveyed. Many scientific reports are divided into six sections: abstract, introduction, experimental, results, discussion, and references and in future lab this format will be used. We will only be concerned with the results, discussion, and perhaps references for this report. Most scientific reports are written in the third person and use few pronouns like I, we, you, etc. **Avoid using personal pronouns.** You may want to include a reference section if you cite sources other than the lab manual. Below is a brief set of guidelines for each of these sections. Please note that it is not the only format used. Be sure to consult the class syllabus, information at the end of experiments in the laboratory manual, and the instructor or teaching assistant with regard to more specific information relating to reports. Remember, your scientific report should be written only by you. Do not do calculations, writing, etc. as a group. Share only the original observations and data with your group members.

**RESULTS:** The focus of this section will be primarily on the synthesis and characterization of the iron salt. All team data must be presented in this section including any values used in the calculations. Be sure to include the following:

1. All of the calculations listed below should have already been done in your lab notebook. Your notebook should have one example using unit cancellation for every type of calculation done. As an appendix to this report, attach copies of any notebook pages with observations, data, graphs, or calculations relating to the iron salt synthesis or analysis to this report. You may want to organize your results using the tables starting on page 2.
2. Synthesis of the iron salt
    a. State any observations during the synthesis.
    b. The mass of iron salt your group measured at various steps in the synthesis.
3. Percent Water
    a. Any Observations.
    b. Percent for each trial.
    c. Median value.
    d. Range and relative percent range.
4. Percent Oxalate
    a. Any Observations.
    b. Results for percent oxalate ion in the iron salt.
    c. A median value.

d. Range and relative percent range for percent oxalate ion trials.
e. Data, median, range, and relative percent range for the molarity of the standard potassium permanganate solution.

5. Percent Iron and Potassium
   a. Any Observations.
   b. A copy of the pH curve.
   c. Percent $K^+$ and $Fe^{+3}$ for the acid-base chemistry extension cation exchange experiment.
   d. If more than one trial was run, list the median value, range, and the relative percent range for each.
5. Show calculations to determine the empirical formula of the iron salt, and give the formula before and after rounding. The theoretical and percent yield can then be calculated using the empirical formula you determined. More on these calculations is provided later in this write-up.

**Synthesis Data**

| | |
|---|---|
| Mass of $K_2C_2O_4 \bullet H_2O$ used (g) | |
| Volume of 0.400g/mL $FeCl_3$ (mL) | |
| Mass of $FeCl_3$ used (g) | |
| Mass of Iron Salt formed (g) *three measured | |
| Observations (color of solutions & crystals) | |

***The mass of the iron salt was measured three times at various points in the purification (see exp. C). If the mass decreased each time what can that show regarding decreased percent yield?**

**Percent by Mass of Water Data**

| | Trial 1 | Trial 2 | Trial 3 | Trial 4 (if done) |
|---|---|---|---|---|
| Mass Empty Beaker | | | | |
| Mass Beaker and Sample | | | | |
| Mass of Sample | | | | |
| Mass after 1st Heating | | | | |
| Mass after 2nd Heating | | | | |
| Mass of Water Lost | | | | |
| % $H_2O$ | | | | |

Median %$H_2O$________________________

Range %$H_2O$ _________________________

Relative % Range for %$H_2O$ ____________

**Percent by Mass of $K^+$ and $Fe^{+3}$ Data**

| | Trial 1 | Trial 2 | Trial 3 (if done) |
|---|---|---|---|
| Mass of Iron Salt Sample | | | |
| Molarity of NaOH | | | |
| Volume of 1st EQ. point | | | |
| Volume of 2nd EQ. point | | | |
| % $K^+$ | | | |
| % $Fe^{+3}$ | | | |

Median %$K^+$________________ Median %$Fe^{+3}$________________

Range of %$K^+$_______________ Range of %$Fe^{+3}$_______________

Relative % Range of %$K^+$_____ Relative % Range of %$Fe^{+3}$_____

**Percent by Mass of $C_2O_4^{2-}$**

| | Trial 1 | Trial 2 | Trial 3 | Trial 4 (if done) |
|---|---|---|---|---|
| Median Molarity $KMnO_4$ | | | | |
| Mass of Sample | | | | |
| Initial Volume | | | | |
| Final Volume | | | | |
| Total Volume | | | | |
| % $C_2O_4^{2-}$ | | | | |

Median %$C_2O_4^{2-}$_________________

Range %$C_2O_4^{2-}$_________________

Relative % Range %$C_2O_4^{2-}$________

**Data Summary**

| | Trial 1 | Trial 2 | Trial 3 | Trial 4 (if done) | Median | Range | Relative % Range |
|---|---|---|---|---|---|---|---|
| % $H_2O$ | | | | | | | |
| % $K^+$ | | | | | | | |
| % $Fe^{+3}$ | | | | | | | |
| % $C_2O_4^{-2}$ | | | | | | | |

Total Median Percent_______

**Empirical Formula of Iron Salt**

| | % $H_2O$ | % $K^+$ | % $Fe^{+3}$ | % $C_2O_4^{-2}$ |
|---|---|---|---|---|
| % of each component | | | | |
| Grams (assuming 100g total) | | | | |
| Molecular Mass of each component | | | | |
| Moles of each component | | | | |
| Unrounded ratio of each component | | | | |
| Rounded ratio of each component | | | | |

Empirical Formula ____________

Molecular Mass______________

**Net Charge for Empirical Formula**

| | $H_2O$ | $K^+$ | $Fe^{+3}$ | $C_2O_4^{-2}$ |
|---|---|---|---|---|
| Charge of ion | | | | |
| Number of ions/molecule | | | | |
| Total charge for each ion | | | | |

Overall Total Charge _____

**Percent Yield of Iron Salt**

| | |
|---|---|
| Actual Yield | |
| Theoretical Yield | |
| % Yield | |

6. If you need help with the calculation of simplest empirical formula, theoretical yield, or percent yield, review the explanation of the calculations provided next and/or ask your TA.
7. Assume the subscript is one for Fe in the formula for the iron salt and round all the other components to whole number for your final reported empirical formula.

## CALCULATIONS: For the iron salt report.

### <u>*Calculating the Empirical Formula for the Iron Salt*</u> *- Do individually.*

*Since you know the % by mass of every component in your iron salt (with reasonable experimental error), the empirical formula can be solved for. Be sure to obtain the percent for oxalate ion the other members of your group obtained. Decide what percent you will use for each of the four components in the iron salt. You will need to explain your logic in the report.*

*First assume that you have a 100 gram sample of green crystals. Then the % of a component, like $K^+$, can be converted into grams. (For example, in a sample where 24.00% exists as $K^+$, the grams of $K^+$ in a 100 g sample is (0.2400) * (100) = 24.00 g. In other words, just swap the units from % to g in a 100 g sample). So,*

$$\% K^+ * 100\ g = \text{grams } K^+ \text{ in a } 100\ g \text{ sample} = 24.00 g\ K^+$$

*The moles of $K^+$ in the 100 g sample can also be determined using potassium's molar mass ($^g/_{mole}$). Be sure to keep four significant figures in your calculations.*

$$mass\ (g) \times \frac{1\ mol}{molecular\ mass\ (g)} = 24.00\ g\ K^+ \times \frac{1\ mol\ K}{39.10\ g\ K} = moles\ of\ K^+\ in\ 100\ g\ sample$$

*Calculate moles for each of the four iron salt components. The moles for each component provide the mole ratio between the components in the formula of the iron salt.*

$$K_{\text{moles of K}}\ Fe_{\text{moles of Fe}}\ (C_2O_4)_{\text{moles of C2O4}} \bullet \text{Moles of } H_2O * H_2O$$

*To simplify this ratio divide by the component with the smallest number of moles. Record these values.*

$$\frac{moles\ of\ K}{moles\ of\ Fe} = number\ of\ K\ atoms\ in\ the\ empirical\ formula$$

*Now round the moles to the nearest whole number. You will want to discuss how much rounding had to be done to get to a whole number in your report. For example, rounding 2.97 to 3 for the empirical formula indicates possible good data while rounding 2.68 to 3 indicates problems with the percent by mass data for one or more of the components.*

### <u>*Calculating the % yield*</u> *– Do individually.*

*The percent yield is calculated by the equation:*

$$\%\ \boldsymbol{yield} = \left(\frac{actual\ yield\ (g)}{theoretical\ yield\ (g)}\right) \times 100\%$$

*The **actual yield** is easy. It is the grams of green crystals made after the synthesis. (Hint: This is in experiment C, after vacuum filtration of the crystals.)*

*The theoretical yield is a little more in depth. It is a calculation that assumes the entire limiting reagent reacts to form product. This is not always true. In the synthesis reaction, $FeCl_3$ was added to a dissolved portion of $K_2C_2O_4 \bullet H_2O$ in the below reaction:*

$$xK^+_{(aq)} + Fe^{3+}_{(aq)} + yC_2O_4{}^{2-}_{(aq)} + zH_2O \rightarrow K_xFe(C_2O_4)_y \bullet zH_2O_{(s)}$$

*We will assume that Fe is the limiting reagent. Therefore, all of the Fe originally in $FeCl_3$ ends up in the product. 16.0 mL of a 0.400 ($^g/_{mL}$) $FeCl_3$ solution was added to the dissolved $K_2C_2O_4 \bullet H_2O$. The grams of $FeCl_3$ is*

$$(16.0\ mL) * (0.400\ ^{g}/_{mL}) = grams\ of\ FeCl_3.$$

*The grams can be converted into moles of $FeCl_3$ by dividing by the molar mass ($^{g}/_{mole}$) of $FeCl_3$.*

$$(grams) \times \left(\frac{1}{molecular\ mass}\right) = mol\ of\ FeCl_3 = mol\ K_xFe(C_2O_4)_y \bullet zH_2O$$

*Since each mole ratio of $FeCl_3$ to $Fe^{+3}$ in solution to iron salt is 1:1:1 (one Fe in iron salt formula):*

$$1\ mole\ of\ FeCl_3 = 1\ mole\ of\ Fe^{+3} = 1\ mol\ of\ K_xFe(C_2O_4)_y \bullet zH_2O$$

*Calculate the molar mass of your iron salt using the empirical formula you determined for the iron salt. Convert the moles of $K_xFe(C_2O_4)_y \bullet zH_2O$ into grams by multiplying by the molar mass:*

$$(moles) * (molar\ mass\ of\ iron\ salt) = g\ of\ K_xFe(C_2O_4)_y \bullet zH_2O \quad (theoretical\ yield)$$

*So in summary using dimensional analysis:*

$$\boldsymbol{Theoretical\ yield\ (g)} = \left|\frac{16.0\cancel{mL\ FeCl_3}}{}\right|\frac{0.400\cancel{g\ FeCl_3}}{1\cancel{mL\ FeCl_3}}\left|\frac{1\ \cancel{mol\ FeCl_3}}{162.0\cancel{g\ FeCl_3}}\right|\frac{1\ \cancel{mol\ salt}}{1\ \cancel{mol\ FeCl_3}}\left|\frac{MM\ salt\ (g)}{1\ \cancel{mol\ salt}}\right|$$

*This is your* ***theoretical yield*** *in grams (If actual yield is larger than the theoretical, recheck calculations or this may indicate a problem with you percent by mass data for one or more of the iron salt components leading to an incorrect empirical formula).*

*Plug the theoretical yield and the actual yield into the percent yield equation.*

$$\%\ \boldsymbol{yield} = \left(\frac{actual\ yield\ (g)}{theoretical\ yield\ (g)}\right) \times 100\%$$

*If the percent yield is low think of what would cause this. Where might have iron salt been lost in the experiment procedure during the synthesis or purification?*

**DISCUSSION:** The discussion section is the most important section of your report. In this section, you must state whether you think your results for the empirical formula are correct or not, and then use your data and results to back this statement up. The significance of your results should be presented to the reader. How did your results compare to the predicted outcome? What do your results mean? If the outcome was not as expected, describe the most probable sources of error. **Be specific! Explaining poor results well is worth more points than poorly explaining good results**. How could the results be improved? This should be the climax of your report so focus on this section! Be sure to use data, results, and equations to clarify and support your conclusions. Some ideas for the discussion:

1. Discuss percent yield. Why is it not 100%?
2. Use the precision in relative percent range for each component to answer the following questions in your discussion. You may want to review the empirical formula problem in assignment 2 done on the first day of lab with a focus on evaluating data (finding bad data).
3. Do all components add up to very close to 100 %? If not, which of the four percentages might be the source of error? Why?

4. Were any problems encountered in rounding to arrive at the final empirical formula? Are the numbers close or far away from whole numbers? What does this tell you? See the example after # 5.
5. Does the charge balance? The overall charge of the iron salt should add up to 0 (neutral). See example below:

***Example:*** *A student analyzed a salt that contained Na, H, and $SO_4$. The percent of each component was found and, like your iron salt, the empirical formula was calculated. The empirical formula after rounding was:*

$$Na_2HSO_4$$

*The student checked his formula by seeing if the charges balanced. Na has a +1 charge, H has a +1, and $SO_4$ has a –2 charge.*

$$2Na + H + SO_4 =$$
$$2(+1) + 1 + (-2) = 1$$

*The student was distraught. Usually a solid has a neutral charge. His didn't. He looked back at the empirical formula before rounding.*

$$Na_{1.55}H_1(SO_4)_{0.92}$$

*H and $SO_4$ were close to one. This was a good indicator that they might be accurate. Neither was rounded very far to get to their whole number. On the other hand, Na was not very close to 2. It had to be rounded up 0.45 units! If this number were rounded down to 1 the charges would balance. Also, he would be rounding down 0.55 units, which was only 0.1 units more than rounding up. So the formula may also be:*

$$NaHSO_4*$$

**Also check the precision of Na to further justify rounding down.*

6. Compare your percent yield to the theoretical yield. Why are they not the same? Propose possible problems with the synthesis that might explain the loss of product yield.
7. Finally, what could you do if you repeated this experiment to improve the results of the synthesis and analysis to determine the formula? Often in a scientific paper this would be called possible areas for "Future Study".

**REFERENCES:** Be sure to cite and list references used to write this report. For example this lab manual should be one of your references. Use the format for references that is most familiar to you.

**APPENDIX – COPY OF NOTEBOOK PAGES:** Make copies of any pages in your lab notebook that contain observations, data, graphs, and calculations relating to Iron Salt Synthesis, Purification, and Analysis.

**GRADING RUBRIC FOR THE IRON SALT REPORT:** Attach the rubric on the following page to the front of your report and fill in your name and the rest of the information at the top of the rubric.

# SYNTHESIS & CHARACTERIZATION OF IRON SALT
# CHM 152L SHORT REPORT GRADING SHEET

STUDENT'S
NAME_________________________________________ Dana ID______________

SECTION LETTER____ DATE_______ INSTRUCTOR____________ TA___________

This sheet should be attached to the front of your report to aid in grading. The report must be typed. Your report should be turned in to your TA

| Pts Earned | Area Graded | Notes |
|---|---|---|
| _____PTS | 10 pts-Overall organization, spelling, grammar, references, report typed | |
| _____PTS | 20 pts-Results (do not discuss results for unknowns) | |
| | ____ Observations and data from experiment C | |
| | ____ Percent water results (each trial, median, & relative percent range) | |
| | ____ Percent $K^+$ and $Fe^{3+}$ (each trial, median, and relative percent range) | |
| | ____ Percent oxalate ion in iron salt results (each trial, median, and relative percent range) | |
| | ____ Calculation simplest empirical formula (list formula before and after rounding) | |
| | ____ Calculation of theoretical and percent yield | |
| | ____ Results presented using tables and correct significant figures | |
| _____PTS | 45 pts-Discussion | |
| | ____ Discuss the formula and its overall charge balance | |
| | ____ Discuss the sum of median percent by mass values for components | |
| | ____ Discuss the rounding needed to get whole numbers formula? | |
| | ____ Relate any possible problems noted above to the precision (expressed as range and relative percent range) of your specific results. | |
| | ____ Focus on specific sources of error related to experimental procedures and data. You will lose points if you use “Human Error” or Machine/Instrument Error” in your discussion! Saying “Calculation Error” without stating specific problems areas could also result in loss of points | |
| | ____ Overall was the argument convincing for why the formula of the iron salt is correct or incorrect (data and precision used in the discussion) | |
| | ____ Comment on percent yield and possible reasons for lower yield | |
| | ____ Possible ways to improve the synthesis or analysis of the iron salt | |
| _____PTS | 5 pts-Appendix (copies of your lab notebook pages with your Fe salt info) | |
| _____PTS | TOTAL out of 80 pts possible for this report. | |