General Comments about the Formal Scientific Report for Experiments 12-090 – 15-015.

The final report will be in the style of a formal paper. The format and content guidelines expected for a scientific report have been outlined previously. There are additional notes outlined below. All reports must be typewritten (preferably using a word processing program) and double-spaced. You should write in the third person past tense rather than the first person or imperative tense. Remember to be consistent with your tenses. You should write as concisely as possible while still reporting all the pertinent information.

The report should include the following:

Cover Page: The cover page should have a title, author and date. You may also want to include your lab section on the cover page.

Introduction: The introduction should clearly state what the paper is reporting. Remember there may be multiple goals and all of these goals should be clearly stated. The introduction should also give some background information (e.g. a description of coordination compounds, what they are, why they are important and how this is related to the work you are reporting in the paper). You should summarize the methodology in your own words). The introduction should include some description of the chemistry being done (i.e. include chemical equations of the reactions that you are reporting). The chemical equations are very helpful for explaining the general procedure used to conduct the synthesis and analysis of your compound. Equations should be set off as separate lines and should be numbered in the right hand margins.

Experimental: This section should include what you actually did in the laboratory. This may deviate from what the manual asked you to do. Since each step in the procedure is reported in sequence, words like then and next are superfluous. You should report masses or volumes of each chemical used and concentration of solutions (to appropriate number of significant figures). You should mention volumetric glassware where appropriate. You need to report important parameters when using equipment (e.g. λ_{max}, vacuum oven temperature, and vacuum oven pressure) Remember it is important to be concise. It is perfectly acceptable in a scientific paper to write: 10.0 mL instead of 10 milliliters and 10 min instead of ten minutes. For example, for the oxalic acid step in the first synthesis you might write: "A solution of 6.1 g (0.048 mol) of oxalic acid dihydrate in 50 mL of water was added to the ferrous ammonium sulfate solution." The experimental section should be divided into subsections using subtitles. For example, the synthesis of the coordination compound can be divided into three subsections with the subtitles, *Preparation of iron (II) oxide, Preparation of* $K_3[Fe(C_2O_4)_3]\cdot 3H_2O$, *Isolation of* $K_3[Fe(C_2O_4)_3]\cdot 3H_2O$, *Preparation of the standard series for determination of iron concentration,* etc.

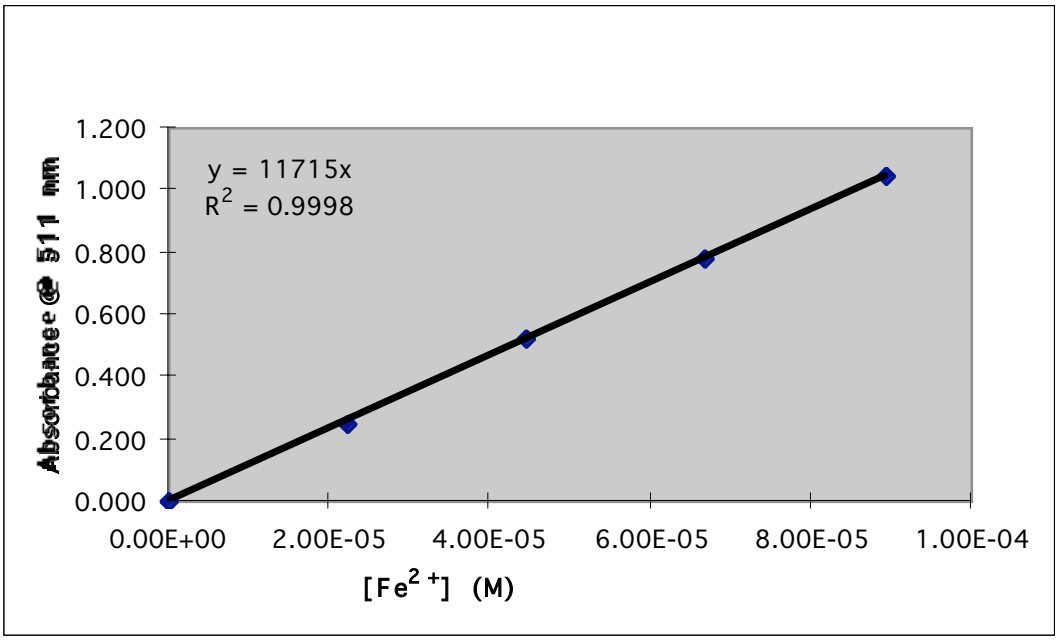
Results: This section should include your experimental observations and a description of your product, actual yield and theoretical yield. You should also identify the limiting reagent. **Raw data** (masses of reagents and products, absorbance measurements, titration volumes, etc.), as well as, **processed data** (calculated masses, moles, concentrations, percentages) should be included in the result section. You should also report your value for ε (with correct units). All data should be presented neatly in tables or figures. All tables and figures should be numbered and have captions which have titles and a clear description of what the figure (or table) depicts. Graphs should have coordinate axes clearly identified and labeled (Remember a graph is a figure so it should also be numbered and have a caption). The result section can be subdivided into the same subsections you used for the experimental section. Some examples of sample tables have been given below. These tables have been provided to give you an idea of how your data (raw and processed) should be presented.

Table 1: Standard Curve Absorbance Data for Fe^{3+}

Aliquot (mL)	Concentration of Fe^{3+} (M)	Absorbance at 511 nm
0.0	0.00	0.000
0.25	2.23×10^{-5}	0.252
0.50	4.46×10^{-5}	0.525
0.75	6.69×10^{-5}	0.780
1.00	8.92×10^{-5}	1.050

The standard series was made using a 8.92×10^{-4} M solution of $[Fe(NH_4)_2(SO_4)_2]\bullet 6H_2O$

Figure 1: Standard Absorbance Curve for Iron (II).



The molar absorbtivity, ε, was determined from the Standard Absorbance Curve for Fe^{2+}

$\varepsilon = 11715\ M^{-1}cm^{-1}$

Table 2: Absorbance Data for $K_3[Fe(C_2O_4)_3]•3H_2O$

Absorbance at 511 nm	Concentration Fe^{2+} (M)	Mass Fe (g)	% Fe
0.744	6.35×10^{-3}	0.0177	10.1
0.764	6.52×10^{-3}	0.0182	10.4
0.868	7.41×10^{-3}	0.0206	11.8
		Average % Fe	10.8
		Standard Deviation (%)	0.909

Table 3: Titration Data for Standardization of $KMnO_4$

Vol. Of $Na_2C_2O_4$ (mL)	Vol. of $KMnO_4$ (mL)*	$[MnO_4^-]$ (M)
5.00	5.16	0.01975
5.00	5.17	0.01971
5.00	5.18	0.01967
	Average $[MnO_4^-]$ (M)	0.01971
	Std. Dev. (M)	4.00×10^{-5}

Concentration of $Na_2C_2O_4$: 0.05100 M

*The blank titration volume of 0.050 mL has been subtracted from each of the titration volumes reported.

Table 4: Titration Data for % Oxalate Determination in $K_3[Fe(C_2O_4)_3]•3H_2O$

Coordination Compound (g)	Vol. of $KMnO_4$ (mL)*	Mole MnO_4^- ($\times 10^{-4}$)	Mole $C_2O_4^{2-}$ ($\times 10^{-4}$)	Mass $C_2O_4^{2-}$ (g)	% $C_2O_4^{2-}$
0.0441	5.47	1.08	2.70	0.0237	53.7
0.0487	6.12	1.21	3.02	0.0266	54.6
0.0471	5.82	1.15	2.87	0.0253	53.7
0.0477	5.92	1.17	2.92	0.0257	53.9
				Average % $C_2O_4^{2-}$	54.0
				Std. Dev. (%)	0.397

*The blank titration volume of 0.050 mL has been subtracted from each of the titration volumes reported.

Table 5: Gravimetric Data for % Water Determination in $K_3[Fe(C_2O_4)_3]•3H_2O$

Mass Crucible (g)	Mass Crucible + Sample before heating (g)	Mass of Sample (g)	Mass Crucible + Sample after heating (g)	% Water in Sample
10.6961	11.7135	1.0174	11.6057	10.60
11.0521	12.0728	1.0207	11.9654	10.52
			Average % Water	10.56
			Std. Dev. (%)	0.0519

Discussion: In this section you will interpret your results. A discussion of standard errors or other estimates of precision should be included here. The empirical formula for the synthesized compound (elemental composition) should be presented in the discussion as well as an explicit comparison of actual and expected % composition for your synthesized compound (you should also include the deduced percentage of potassium for your compound and compare it with the expected percentage of potassium). A brief, logical explanation of why your experimentally determined % composition values differ from the actual % composition should be included. You should include a discussion of confidence limits (see following pages). A final conclusion should be drawn – Did you or did you not make what you set out to synthesize?

References: This section is not optional in a scientific paper unless everything you did was absolutely original. You should reference any and all literature sources that you used, if you refer to them explicitly in your report. You should include appropriate references at the end of the paper and include citation marks in the body of the paper. At the very minimum you should cite your text and your lab manual. You should assign a number to each citation based on the order in which you cite them in your report. If you cite the exact same source more than once in your report use the original. The number should follow the text. Use either superscript or parenthesis. List the sources at the end of the report as end notes and order the citations numerically. There are several acceptable formats for references. One very commonly used by chemists is the ACS format:

1. For journal articles: authors' surnames and initials, abbreviated journal title (italicized or single-underlined), year (bold-faced or double-underlined), volume (italicized or single-underlined), inclusive page numbers.

   Mann, C.J.; Weiner, H. *Prot. Sci.* **1999**, *8*, 1922-1929.

2. For books: authors' surnames and initials, book title ((italicized or single-underlined), edition; publisher: city, year; chapter or inclusive page numbers.

   Zumdahl, S.S., *Chemistry*, 7th ed.; Houghton Mifflin: New York, 2007; p. 946.

3. For a book with an editor and no author: editor's surname, first name and middle initial, ed. book title. (italicized or single-underlined), city: publisher, year.

   Chanatry, Julie A., *Chemistry 102 Laboratory Manual*. Hamilton, NY: Colgate University Printing, 2008.

If you refer multiple times to the same citation in one report, do not repeat it in your reference list; reuse the superscript number with which it first appears in your paper.

Using Confidence Limit Data to Evaluate Elemental Analysis

Now that you have analyzed the elemental makeup of your product, you want to use the data from the analysis to show that you have indeed synthesized the compound, $K_3[Fe(C_2O_4)_3]\bullet 3\ H_2O$. There is no way, by looking at the data, that we can know whether we did the experiment correctly. What we can do, however, is use the standard deviation to calculate confidence limits – a range within which the true value of the measured quantity should occur with a specified probability.

For a *large* number of measurements, the true value of x will be within the range $x \pm s$ for 68 per cent of the time, within the range $x \pm 1.96s$ for 95 per cent of the time. To be 99% certain that the correct answer is within a given range, we need to consider the range $x \pm 2.58s$. In this experiment, we did not perform a "large" number of experiments, so we need to have some way of estimating our confidence for a smaller (less than infinite) number of experiments. To do this we introduce the variable, *t*. You can think of *t* as giving us some measure of the best we can do under the circumstances. Table 1 lists some factors that can be used for calculating confidence limits; as expected, these factors vary with n and the degree of confidence desired.

Table 1: Factors for Calculating Confidence Limits

Confidence Limit	**80%**	**90%**	**95%**	**99%**	**99.5%**
	$t/\sqrt{N}$	$t/\sqrt{N}$	$t/\sqrt{N}$	$t/\sqrt{N}$	$t/\sqrt{N}$
N					
2	2.17	4.46	8.98	45.0	450.
3	1.09	**1.69**	2.48	5.72	18.2
4	0.82	**1.18**	**1.59**	2.92	6.45
5	0.68	0.95	1.24	2.06	3.84
6	0.60	0.82	1.05	1.65	2.80
7	0.54	0.74	0.93	1.40	2.25
8	0.50	0.67	0.83	1.24	1.91
9	0.47	0.62	0.77	1.12	1.68
10	0.44	0.58	0.71	1.02	1.51

By using the expression:

$$\mu = \overline{X} \pm {}^{t}\!/_{\sqrt{N}} \bullet s$$

(where μ is the degree of confidence required, $\overline{X}$ is the mean, $t/\sqrt{N}$ is the confidence factor from Table 1, *N* is the number of replicates and s is the standard deviation) we generate a range of values.

For example, in determining the percentage of water in your compound, say you found that the mean percentage of water and standard deviation of your sample is 10.3 ± 0.6 % for 3 trials. To be 90% certain that the true value lies within our range of values, we can do the following calculation:

$$10.3\ \% \pm (1.69)(0.6\ \%) = 10.3 \pm 1.0$$

This means that you can be 90% sure that your experimentally determined value lies in the range between 9.3% – 11.3%.

You should now compare your experimentally determined percentage of water with the theoretical percentage of water in $K_3[Fe(C_2O_4)_3]\bullet 3\ H_2O$:

$$(3 \text{ mole } H_2O/1 \text{ mole } K_3[Fe(C_2O_4)_3]\bullet 3\ H_2O) \times 100\%$$
$$((3 \times 16.0 \text{ g/mole})/491.26 \text{ g/mole}) \times 100\ \% = 11.0\ \%$$

Since the theoretical percentage of water in $K_3[Fe(C_2O_4)_3]\bullet 3\ H_2O$ also lies in the range between 9.3% - 11.3% you can say that you **cannot** be sure at the 90% confidence limit that the experimental percentage is **different** than the theoretical percentage. (The 90 % or 95 % confidence limits are good choices for making these determinations). This is good – you are confident that there is no difference between experimental and theoretical percentage of water. This supports the idea that you did synthesize $K_3[Fe(C_2O_4)_3]\bullet 3\ H_2O$.

Let us look at another example, if the percentage of iron in a sample was found to be 10.5 ± 0.5 % for four trials. You can be 90% confident that the actual value will be in the range of 9.91% to 10.09% (10.5 % ± (1.18)(0.5)%). The theoretical percent of iron in $K_3[Fe(C_2O_4)_3]\bullet 3\ H_2O$ is 11.4 %. The theoretical value does not fall in the 90 % confidence limit range of your experimental data, in fact your experimental value appears to be lower than the theoretical value. You can say that you are 90 % confident that the percent iron is actually **lower** than the theoretically predicted amount.

You can increase the range of certainty for your experimentally determined percent of iron (i.e., look at the 99 % CL). In this case, the calculated range **does** encompass the theoretical percentage of iron. (you should do the calculations to convince yourself). At the 99% confidence limit you cannot be sure that the experimentally determined percentage of iron is actually lower than the theoretically predicted amount. Thus you can say you are between 90-99 % confident that your compound contains less iron than that expected.

In this case, you should then attempt to explain why your experimentally determined value may be lower than the theoretical predicted amount (think about what you measured in the experiment and where errors could have been introduced[1]).

You should also do this type of analysis for the oxalate analysis. Remember use your data.

[1] Human error is not an acceptable explanation – citing human error basically tells the reader that you did not do the work carefully and could call into question the reliability of all of your data.