#### **FULL REPORT GUIDE**

#### General Chemistry Lab Reports

Full reports combine the introduction, methods, results and discussion sections in a single coherent paper. Full reports for general chemistry will also include a title page at the beginning and a brief conclusion following the results and discussion section. Full reports for other chemistry courses may also require an abstract and expanded conclusions section. Every section of the full report will follow the American Chemical Society formatting rules.<sup>1</sup>

When preparing a title page, include the following, each on their own line, centered:

- 1. Title of the experiment. Capitalize according to the ACS Style Guide.<sup>1</sup>
- 2. Name(s).
- **3.** Course information. Lab day and time, instructor (e.g. Monday 1:00 5:00, Mrs. Holmes)
- 4. Affiliation. Azusa Pacific University, Department of Biology and Chemistry, Azusa, CA

Appropriate format for title page: legible font size 14, with 1.5 spacing, 1 inch margins

Additional details for the full report:

- The full report for general chemistry should be in the order: title page, introduction, methods, results and discussion, conclusion, references. An error analysis should be included in the discussion section.
- Each section should be labeled with a section header in all caps and bolded (e.g. **INTRODUCTION**). The sections should be included continuously after each other, without a page break between them.
- Tables and figures should be placed in the text, after the paragraph where they are first referenced. Figures should be half-page width or larger.
- Any additional data (such as the raw data that makes up the figures) may be attached to the report in an appendix
- A brief (2-3 sentences) conclusion should be included after the results and discussion section, under the header **CONCLUSION**. The conclusion should include:
  - o Restatement of the hypothesis
  - o Explanation of whether the results support or refute the hypothesis
  - Explanation of the overall suitability of the method to investigate the hypothesis (this conclusion is made from the discussion of the data and the error analysis)
- References are listed at the end of the report, in the order they are cited in the text of the report, under the header **REFERENCES**
- Appropriate format for entire report: legible font size 12, with 1.5 spacing, indented paragraphs, 1 inch margins, page numbers in top right, and academic language.

<sup>1.</sup> The ACS Style Guide; Dodd, Janet S. Ed.; American Chemical Society; Washington, DC, 1997.

## Sample Full Report for Spectrophotometric Analysis of Aspirin

# Spectrophotometric Analysis of Aspirin Carrie Miller

Monday 1:00 – 5:00, Mrs. Holmes

Azusa Pacific University, Department of Biology and Chemistry, Azusa, CA

#### INTRODUCTION

Commercial pain relievers containing acetylsalicylic acid (the active ingredient in aspirin) were used regularly by 43 million adults in the United States in 2010, an increase of 57% from 2005. Given the ubiquity of these pain relievers, it is important that the amount of acetylsalicylic acid in pain reliever tablets match the amount given by the manufacturer. The purpose of this experiment is to determine the mass of acetylsalicylic acid in a commercial aspirin tablet. Because drug manufacturing is overseen by the Food and Drug Administration in the United States, it is hypothesized that the amount of acetylsalicylic acid measured will be within 5% of the amount given on the label.

The mass of acetylsalicylic acid present in the tablets was determined using spectrophotometric analysis. Because acetylsalicylic acid is colorless, it was hydrolyzed to form salicylate, which in turn was reacted with the chromophoric reagent  $Fe(H_2O)_6^{3+}$  to form the chromophore.<sup>2</sup> A chromophore is a colored compound that absorbs light with a certain wavelength. Because acetylsalicylic acid and the chromophore have a one to one molar ratio, the amount of chromophore detected is directly proportional to the amount of acetylsalicylic acid present. A calibration curve was generated from a standard solution of acetylsalicylic acid. This curve was fit with Beer's Law,

$$A = \varepsilon lc \tag{1}$$

where A is the absorbance (arbitrary units),  $\varepsilon$  is the molar absorptivity (concentration/cm), 1 is the path length of the cuvette (cm), and c is the concentration of the iron(III)salicylate chromophore. Beer's Law is the theory that connects the absorbance of light by a colored solution with the concentration of the chromophore.

#### **METHODS**

Part A: Beer's Law

A calibration curve for acetylsalicylic acid (ASA) was generated using a Barnstead Turner SP-350 Plus spectrophotometer. A 0.104 g sample of ASA was hydrolyzed by heating it in 10.0 mL of a 1.0 M NaOH solution. In order to develop the iron(III) salicylate chromophore complex, five samples of the hydrolyzed ASA solution (0.1 mL, 0.2 mL, 0.3 mL, 0.4 mL, and 0.5 mL, respectively) were diluted to 25 mL in 0.020 M iron(III) chloride. The absorbance and transmittance of the five solutions were measured at 530 nm.

## Part B: Analysis of aspirin in a commercial pain reliever

The mass of ASA in commercial pain reliever tablets was determined using spectrophotomeric analysis. Two tablets (from the same container) were separately ground by hand and each hydrolyzed by heating in 10 mL of 1.0 M NaOH. For each tablet, 0.1 mL and 0.2 mL of the hydrolyzed compound solution were diluted to 25 mL in 0.020 M iron(III) chloride. The absorbance and transmittance of the solutions were measured at 530 nm.

#### **RESULTS AND DISCUSSION**

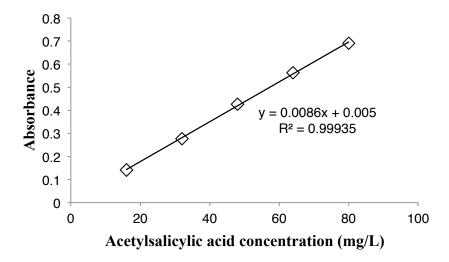
The molar absorptivity of acetylsalicylic acid, as determined by a linear fit of the calibration curve date in Figure 1, is 0.0864 L/mg\*cm. The large R² value (0.99935) shows that the data used to generate the calibration curve is very linear, and is therefore well described by Beer's law. The calibration curve was used to determine the concentration (and thus mass) of acetylsalicylic acid in two tablets, diluted to two different concentrations (Table 1). The two tablets analyzed were only 1% different in mass, but had an average of 8% difference in the mass of acetylsalicylic acid present. The average mass of acetylsalicylic acid in the tablets is 317 mg. The mass of acetylsalicylic acid listed on an aspirin bottle is 325 mg, giving a small difference (2.46%) between the measured value and the amount given on the bottle. Because this percent difference is less than 5%, the two numbers are considered to be in good agreement with each other.

**Table 1.** Physical and spectroscopic properties of aspirin tablets

Quantity	Tablet 1	Tablet 2
Mass of tablet (g)	0.375	0.370
Absorbance, 0.4 mL dilution	0.484	0.424
Transmittance, 0.4 mL dilution	32.7%	37.7%
Absorbance, 0.6 mL dilution	0.657	0.636
Transmittance, 0.6 mL dilution	22.0%	23.1%
Mass acetylsalicylic acid, 0.4 mL dilution (mg)	346	303

**Table 1.** Physical and spectroscopic properties of aspirin tablets

Quantity	Tablet 1	Tablet 2
Mass acetylsalicylic acid, 0.6 mL dilution (mg)	314	304



**Figure 1.** Beer's Law calibration curve for the absorbance of 530 nm light by acetylsalicylic acid.

The experimental design includes method errors that affect the average mass of aspirin determined to be in the tablets. First, only two tablets were used to for the trials. This small sample size increases the possibility of indeterminate errors affecting the results. To reduce this error, additional tablets would need to be tested.

Second, both the hydrolysis reaction of acetylsalicylic acid with sodium hydroxide and the reaction of salicylic acid with iron(III) are assumed to proceed to completion. This assumption is reasonable because the NaOH and iron(III) solutions were present in excess compared to the molar amount of acetylsalicylic acid. If the reactions did not proceed to completion, however, the amount of acetylsalicylic acid present in the tablet would be underestimated by this technique. The direction of error caused by incomplete reactions is consistent with the result of a lower measured value of acetylsalicylic acid compared to the value on the bottle.

## **CONCLUSION**

This experiment determined the amount of acetylsalicylic acid in an aspirin tablet, which was hypothesized to be the same as the amount on the bottle. A difference of 2.46% between the amount measured and the amount on the bottle supports this hypothesis. A variety of factors support the suitability of this experiment to measure the amount of acetylsalicylic acid in a tablet: the reaction to create the colored chromophore proceeded to completion (or near completion), and the calibration curve generated with standard solutions of the chromophore was fit to a linear equation (Beer's law), which allows for a precise determination of the amount of acetylsalicylic acid in the unknown.

#### REFERENCES

- 1. Zhou, Y. J.; Boudreau, D. M.; Freedman, A. N. Trends in the use of aspirin and nonsteroidal anti-inflammatory drugs in the general US population. *Pharmacoepidemiology and Drug Safety* **2014**, *23*, 43-50.
- 2. White, J. and Holmes, R. *CHEM 151/152 General Chemistry Laboratory Manual Azusa Pacific University: Azusa, CA, 2013.*

#### **INTRODUCTION GUIDE**

General Chemistry Lab Reports

The introduction section of a lab report provides the background and significance of the experiment.

When writing an introduction section, include the following:

- 1. **Hypothesis**. Describe what prediction is being tested by the experiment (that is, answer the questions, "what is the purpose of the experiment?" and "what result do you expect to get, and why?")
- **2. Motivation**. Answer the question, "why would a scientist care about the outcome(s) of the experiment?"
- **3. Background.** Explain the concepts needed to understand the experiment.
  - The background should include *two or more* references to outside sources (one of which may be the lab manual). Primary sources like journal articles (accessed through the library website) are suitable. Acceptable secondary sources are the lab manual and course textbook. Other secondary sources like online encyclopedias (e.g. Wikipedia) are unacceptable. The information obtained through a source should be cited in text by a superscript number at the end of the sentence. The full reference is given at the end of the report, after the conclusion. Use the American Chemical Society reference style.<sup>a</sup>
  - Mathematical and chemical equations should be set apart, centered in its own line. Equations should be sequentially numbered; the numbers should be right-aligned and enclosed in parentheses. A description of each variable (including units if applicable) should follow a mathematical equation. Chemical structures should be labeled with the chemical name in a chemical reaction.
- **4. Experimental design.** Provide a brief (1-2 sentence) rationale of why the experiment will be suitable for testing the hypothesis.
- **5.** Appropriate format: legible font size 12, with 1.5 spacing, indented paragraphs, 1 inch margins, page numbers in top right if longer than a page, and academic language.
- **6.** Include your name, course, lab instructor, and date in the top left corner in font size 10 with no spacing. (Note: full formal reports will have a title page instead.)

When writing the introduction, avoid the use of the first person. For any content that is currently known to be true (and expected to be true in the future), use present tense. For any content that refers to a procedure or method that has already been performed, use past tense.

a. The ACS Style Guide; Dodd, Janet S. Ed.; American Chemical Society; Washington, DC, 1997.

## Sample Introduction for Spectrophotometric Analysis of Aspirin

Commercial pain relievers containing acetylsalicylic acid (the active ingredient in aspirin) were used regularly by 43 million adults in the United States in 2010, an increase of 57% from 2005. Given the ubiquity of these pain relievers, it is important that the amount of acetylsalicylic acid in pain reliever tablets match the amount given by the manufacturer. The purpose of this experiment is to determine the mass of acetylsalicylic acid in a commercial aspirin tablet. Because drug manufacturing is overseen by the Food and Drug Administration in the United States, it is hypothesized that the amount of acetylsalicylic acid measured will be within 5% of the amount given on the label.

The mass of acetylsalicylic acid present in the tablets was determined using spectrophotometric analysis. Because acetylsalicylic acid is colorless, it was hydrolyzed to form salicylate, which in turn was reacted with the chromophoric reagent Fe(H<sub>2</sub>O)<sub>6</sub><sup>3+</sup> to form the chromophore.<sup>2</sup> A chromophore is a colored compound that absorbs light with a certain wavelength. Because acetylsalicylic acid and the chromophore have a one to one molar ratio, the amount of chromophore detected is directly proportional to the amount of acetylsalicylic acid present. A calibration curve was generated from a standard solution of acetylsalicylic acid. This curve was fit with Beer's Law,

$$A = \varepsilon lc \tag{1}$$

where A is the absorbance (arbitrary units),  $\varepsilon$  is the molar absorptivity (concentration/cm), l is the path length of the cuvette (cm), and c is the concentration of the iron(III)salicylate chromophore. Beer's Law is the theory that connects the absorbance of light by a colored solution with the concentration of the chromophore.

<sup>1.</sup> Zhou, Y. J.; Boudreau, D. M.; Freedman, A. N. Trends in the use of aspirin and nonsteroidal anti-inflammatory drugs in the general US population. *Pharmacoepidemiology and Drug Safety* **2014**, *23*, 43-50.

<sup>2.</sup> White, J. and Holmes, R. *CHEM 151/152 General Chemistry Laboratory Manual Azusa Pacific University: Azusa, CA, 2013.* 

#### **METHODS GUIDE**

#### General Chemistry Lab Reports

The procedure for an experiment is written in the methods section of a lab report. This section is written as a narrative rather than a step-by-step list of the procedure.

When writing a methods section, include the following:

- 1. Explanation of methods used, in enough detail that another scientist could replicate your experiment (DO NOT copy the procedure from the manual!). In a lab report, you will generally include more detail than is typically seen in a research article.
- 2. Chemicals used, including their concentrations, masses, and/or volumes, depending on how they were measured
- 3. Variables measured (including the number of trials)
- 4. Instrument or apparatus used
- 5. DO NOT say "you recorded the data in your notebook." Recording data and observations is implied and should not be explicitly stated.
- 6. Appropriate format: legible font size 12, with 1.5 spacing, indented paragraphs, 1 inch margins, page numbers in top right if longer than a page, and academic language.
- 7. Include your name, course, lab instructor, and date in the top left corner in font size 10 with no spacing. (Note: full formal reports will have a title page instead.)

It is important that the methods reflect what was actually done in the experiment rather than the written procedure. This section is written in past tense, using passive voice. Avoid using first person pronouns.

#### Sample methods for Spectrophotometric Analysis of Aspirin

Part A: Beer's Law

A calibration curve for acetylsalicylic acid (ASA) was generated using a Barnstead Turner SP-350 Plus spectrophotometer. A 0.104 g sample of ASA was hydrolyzed by heating it in 10.0 mL of a 1.0 M NaOH solution. In order to develop the iron(III) salicylate chromophore complex, five samples of the hydrolyzed ASA solution (0.1 mL, 0.2 mL, 0.3 mL, 0.4 mL, and 0.5 mL, respectively) were diluted to 25 mL in 0.020 M iron(III) chloride. The absorbance and transmittance of the five solutions were measured at 530 nm.

Part B: Analysis of aspirin in a commercial pain reliever

The mass of ASA in commercial pain reliever tablets was determined using spectrophotomeric analysis. Two tablets (from the same container) were separately ground by hand and each hydrolyzed by heating in 10 mL of 1.0 M NaOH. For each tablet, 0.1 mL and 0.2 mL of the hydrolyzed compound solution were diluted to 25 mL in 0.020 M iron(III) chloride. The absorbance and transmittance of the solutions were measured at 530 nm.

#### RESULTS AND DISCUSSION GUIDE

General Chemistry Lab Reports

Data from an experiment are presented in the results and discussion section of a lab report. Data can be presented in tables or figures (graphs). Tables are used to compare a relatively small number of data points, whereas figures are generally used to highlight trends in larger sets of data. In the general chemistry lab, tables and figures will be prepared using the American Chemical Society formatting rules. Instructions on how to prepare figures and tables with the correct format are in the general chemistry lab manual.<sup>2</sup>

When preparing a table, include the following:

- 1. Title. Every table must have a brief, descriptive title that summarizes the table's purpose in a non-sentence format. Titles go at the top of the table.
- **2.** Column headers. Every column must have a very short header (one or two words, typically), including units (in parenthesis).

When preparing a figure, include the following:

- 1. Caption. Every figure must have a descriptive caption that summarizes the figure's purpose in a non-sentence format. Captions go at the bottom of the figure.
- **2. Axis labels**. Each axis must be labeled with its appropriate variable (first letter capitalized), units (in parenthesis), and scale. The axes should extend just past the data.
- **3. Symbols**. Plot all data as xy scatter plots unless told to do otherwise. Do not include a line connecting the symbols. If there is more than one set of data in the same figure, each data set should be given a unique symbol and a legend should be included to denote which data set goes with which symbol.
- **4.** Trend line. Add a trend line to the data set(s). Below the figure legend, include the trend line equation(s) with the R<sup>2</sup> value(s).

The results of an experiment are interpreted in the discussion section. This part makes up the narrative text that accompanies the tables and figures. Avoid the use of first person pronouns. Present tense is used for interpretation of the data. Any references to the experimental procedure should be in past tense.

When writing a discussion, include the following:

- 1. You must reference and explain every table and figure. When referencing the tables and figures specifically, use "Table X" and "Figure X."
- 2. If you are reporting a large set of data, give the significance of any trends observed in the data (i.e., is variable A directly or inversely related to variable B, and what does their relationship mean?)
- 3. If you are comparing different groups, clarify the differences. That is, do not simply say "group 1 and group 2 are different" explain *how* they are different. Include the direction of difference (which group is larger or smaller) and the percent difference between them.
- 4. The relationship of your results to your original hypothesis (from your introduction). This statement serves as the conclusion of the discussion.
- 5. Appropriate format: legible font size 12, with 1.5 spacing, indented paragraphs, 1 inch margins, page numbers in top right if longer than a page, and academic language.
- 6. Include your name, course, lab instructor, and date in the top left corner in font size 10 with no spacing. (Note: full formal reports will have a title page instead.)

<sup>1.</sup> The ACS Style Guide; Dodd, Janet S. Ed.; American Chemical Society; Washington, DC, 1997.

<sup>2.</sup> White, J. and Holmes, R. CHEM 151/152 General Chemistry Laboratory Manual Azusa Pacific University: Azusa, CA, 2013

## Sample Results and Discussion for Spectrophotometric Analysis of Aspirin

**Table 1.** Physical and spectroscopic properties of aspirin tablets

Quantity	Tablet 1	Tablet 2
Mass of tablet (g)	0.375	0.370
Absorbance, 0.4 mL dilution	0.484	0.424
Transmittance, 0.4 mL dilution	32.7%	37.7%
Absorbance, 0.6 mL dilution	0.657	0.636
Transmittance, 0.6 mL dilution	22.0%	23.1%
Mass acetylsalicylic acid, 0.4 mL dilution (mg)	346	303
Mass acetylsalicylic acid, 0.6 mL dilution (mg)	314	304

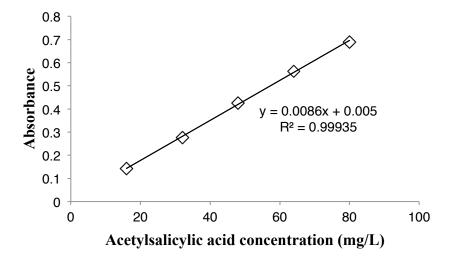


Figure 1. Beer's Law calibration curve for the absorbance of 530 nm light by acetylsalicylic acid.

The molar absorptivity of acetylsalicylic acid, as determined by a linear fit of the calibration curve date in Figure 1, is 0.0864 L/mg\*cm. The large R² value (0.99935) shows that the data used to generate the calibration curve is very linear, and is therefore well described by Beer's law. The calibration curve was used to determine the concentration (and thus mass) of acetylsalicylic acid in two tablets, diluted to two different concentrations (Table 1). The two tablets analyzed were only 1% different in mass, but had an average of 8% difference in the mass of acetylsalicylic acid present. The average mass of acetylsalicylic acid in the tablets is 317 mg. The mass of acetylsalicylic acid listed on an aspirin bottle is 325 mg, giving a small difference (2.46%) between the measured value and the amount given on the bottle.

#### **ERROR ANALYSIS GUIDE**

General Chemistry Lab Reports

In formal report writing, an error analysis discusses the validity and reliability of experimental results. An error analysis is placed between the results and discussion section and the report's conclusion. This allows the author to present the experimental results, discuss the validity of those results, and then use that information to reach a sound scientific conclusion.

When writing an error analysis, include the following:

- 1. Comparison of experimental results to the accepted or theoretical answer. A percent error, percent difference, percent recovered, etc. should be included in this section.
- 2. Statement of whether or not that amount of deviation was acceptable. The lab instructor will inform students as to the acceptable range for each experiment.
- 3. List of 2-3 errors that actually occurred in your experiment.
  - a. Only report a gross error if it actually occurred and your lab instructor determined there was not enough time to repeat the experiment.
  - b. Indeterminate error is discussed when only ONE trial is performed. This is because random error is reduced by averaging results from many trials. So, if you have only one trial, no averaging occurs.
  - c. It is best to focus on method errors, which are assumptions in the experiment. Review the lab manual, your notebook pages, and report sheet questions as these may reveal assumptions in experiments.
- 4. Link each error to its effect on your final results. In other words, how would the errors (step 3) cause the deviation of your final answer (steps 1 & 2)? Refer to the mathematics when appropriate, but do so in words using "directly proportional" and "indirectly proportional." Whenever appropriate, explain if the errors cancel each other out.
- 5. Explain briefly how to reduce these errors in future experiments.
- 6. Appropriate format: legible font size 12, with 1.5 spacing, indented paragraphs, 1 inch margins, page numbers in top right if longer than a page, and academic language.
- 7. Include your name, course, lab instructor, and date in the top left corner in font size 10 with no spacing. (Note: full formal reports will have a title page instead.)

#### Sample Error Analysis for A Series of Chemical Reactions (General Chemistry I)

In the series of chemical reactions, 96.2% of the copper metal was recovered. This is within the acceptable range for experiment #2, therefore, no significant gross error occurred. However, method error was present throughout the procedure.

First, it was assumed that each reaction was driven to completion; however, it is quite possible that some copper did not react and was left behind. This would lead to a lower percent recovery. Relying on the human eye to determine the completion of a reaction is an unavoidable limitation of the experimental procedure.

Secondly, the experiment assumed that copper containing solutions and solids could be transferred without loss; however, it was clear that copper was lost in these transfers and particularly on the pH and filter papers. The best way to reduce this error is to reduce the total number of transfers and to rinse equipment and papers thoroughly. The washings, which contain copper, would be collected and continued in the experiment. It would also be important to dip the pH paper as minimally as possible into the solution to reduce the loss of copper in that step.

Lastly, the experiment assumed that zinc solid would completely dissolve in reaction 5. Zinc however was clearly present in our final product. As zinc weighs more than copper, the contamination of zinc would significantly increase the percent recovered. To reduce this error, additional acid is used and a longer time is necessary for the reaction to reach completion.

These method errors are heading in opposing directions to increase and lower the percent recovery of copper. Therefore, the errors cancel each other out. This is the only reason why our final mass of copper was so close to 100% recovery when the experiment was full of error.

### Sample Error Analysis for Spectrophotometric Analysis of Aspirin (General Chemistry II)

The amount of acetylsalicylic acid measured in the experiment was 2.46% lower than the value on the aspirin bottle. Because this percent difference is less than 5%, the two numbers are considered to be in good agreement with each other.

The experimental design includes method errors that affect the average mass of aspirin determined to be in the tablets. First, only two tablets were used to for the trials. This small sample size increases the possibility of indeterminate errors affecting the results. To reduce this error, additional tablets would need to be tested.

Secondly, both the hydrolysis reaction of acetylsalicylic acid with sodium hydroxide and the reaction of salicylic acid with iron(III) are assumed to proceed to completion. This assumption is reasonable because the NaOH and iron(III) solutions were present in excess compared to the molar amount of acetylsalicylic acid. If the reactions did not proceed to completion, however, the amount of acetylsalicylic acid present in the tablet would be underestimated by this technique. The direction of error caused by incomplete reactions is consistent with the result of a lower measured value of acetylsalicylic acid compared to the value on the bottle.