EXPERIMENT 3: Introduction to Analytical Technical Writing: USING analytical chemistry journal publications format for laboratory reports

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PURPOSE

Familiarize the student with the fundamental aspects of analytical technical writing. At the end of this exercise, the student should know the key criteria used to write a scientific report or manuscript and implement that knowledge to write their own reports according to the Analytical Chemistry and American Chemical Society (ACS) guidelines.

THEORY

Research articles are devoted to disseminate new and original knowledge. Therefore, articles in analytical chemistry may address the general principles of chemical measurement science and need not directly address existing or potential analytical methodology. Articles may be entirely theoretical with regard to analysis or may report experimental results that bear on theory. Articles may contribute to any of the phases of analytical operations, including sampling, chemical reactions, separations, instrumentation, measurements, and data processing. Articles dealing with known analytical methods should offer either a significant, original application of the method, or a significant improvement of the method, or of results for an important analyte.

Writing reports in the analytical laboratory incorporate the same basic structure and style of the reports published in the journal of Analytical Chemistry. The journal has the highest impact factor in the field and is one of the major publications issued by the ACS. In this exercise, students will learn the basic structure of an analytical manuscript named: abstract, introduction, experimental procedure, results and discussion, conclusion and supporting information. In the scientific community, clarity and precision are vital to the purpose of scientific writing, which is intended to report new theories and findings so they can be used and tested. Therefore, an understanding the role of each section and organizing them in a coherent scientific style is one of the most important goals in the course.

References:

1. Cetin, S.; Hackam, D. J. An Approach to the Writing of a Scientific Manuscript. *J. Surg. Res.*. **2005,** *128*, 165–167.
2. http://pubs.acs.org/paragonplus/submission/ancham/index.html; ACS, ―Instructions to Authors for Analytical Chemistry‖
3. Tischler, M. E. *Scientific Writing Booklet;* University of Arizona [Online], http://www.biochem.arizona.edu/marc/Sci-Writing.pdf.

PRELABORATORY EXERCISE

1. Read this exercise, and using the guidelines provided, write a new title and abstract for your first laboratory experience (Experiment 1).
2. Using the UPRM Library Databases (Science Direct or ACS Journals), find at least 2 references from recent, peer reviewed journals (2005-present) under the topic of ―Instrument Validation‖. Cite them using the Analytical Chemistry journal referencing format. Bring an electronic copy of them to the class.
3. Using those articles, comment briefly on how other investigators determine the performance of an instrument validation.
4. Using the provided guidelines prepare an outline for a full report of your first laboratory experience.
5. Write a brief introduction (<2 pages) under the topic: ―Validation of an analytical balance‖ with citations from current literature.

APPARATUS AND MATERIALS

- Data collected in Experiment 1: Exercise 1-Calibration of the Analytical Balance.
- Computer with Windows Vista or XP-Professional,MS Office and a mass storage device (i.e. jump drive, CD-R). **Students are encouraged to bring their personal computer to the lab for this exercise.**

EXERCISE

Objective: ***To prepare a Full-Laboratory Report for the Exercise Spectrophotometric Analysis of Aspirin, using the ACS publications format described in this manual and the following guidelines***

I. Basic Guidelines:

1. **Word usage in scientific writing:** In general, the best writing is simple and direct. Writing that is straightforward and thorough is most easily understood. It also important to avoid the temptation of using:
    - more words than necessary
    - a complicated word if a simpler one will do just as well

    A number of authors seem to perceive that writing in a complicated way makes their arguments sound more serious, scholarly and convincing. While this type of writing may sound serious, it is no more trustworthy than a writing that is simple and direct. Instead, it may sound pretentious, arrogant and certainly, it is more difficult to understand. Writers should also consider the following:
    - Use a US-English spell checker as aid with the grammar, syntax, punctuation and spelling of your manuscript.
    - Use words according to the meaning understood by the average person.
    - Avoid speculations and unnecessary overstatements.
    - Always write in third person.

2. **Prepare an outline of your manuscript:**
    - Determine the:
        - purpose of your paper
        - audience
    - List all the ideas that you want to include in your paper:
        - summarize the problem(s)
        - key elements pertaining to problem(s)
    - Organize your findings in subsections from general to specific

3. **Grammar:** In scientific or technical writing, the authors are not really as important as the process or principle being described. Therefore instead of writing ***"I adjusted the flow rate to 20 mL/min for the entire analysis"***, the author would write ***"The analysis was conducted at a constant flow rate of 20 mL/min."*** Thus in scientific writing, the passive voice is often preferred to indicate objective procedures. Scientist and engineers are interested in analyzing data and in performing studies that other researchers can replicate.

II. Specific Guidelines:

1. **Title:** Often the first, and possibly the only part read by colleagues. It must communicate the most important concepts of the research including the key scientific finding, the methods used to conduct the study, and the target system under study. A carefully composed title that captures the interest of the audience typically:
    - consists of a meaningful sentence fragment no more than two lines or ~15 words long
    - encourages the audience to read the abstract and the remainder of the report

- aids in the computer automated searching of scientific databases

The following are some representative titles for Gas Chromatographic applications found in the scientific literature.

- ***Microbial Metabolomics with Gas Chromatography/Mass Spectrometry***
- ***Quantitative Analysis and Structure Determination of Styrene/Methyl Methacrylate Copolymers by Pyrolysis Gas Chromatography***
- ***Headspace Analysis of Engine Oil by Gas Chromatography/Mass Spectrometry***

2. **Abstract:** Provide a concise and self-contained summary of the report, allowing a reader to quickly determine the nature and scope of the entire research report. It expands the title by briefly summarizing the problem, methods employed to conduct the study, key scientific findings, and main conclusions. Since the abstract is a condensed and focused summary of the report, it should only provide information that is included elsewhere in the manuscript. The abstract should:
   a. consists of a single paragraph containing approximately 80 - 200 words
   b. not cite references, tables, or figures or refer to sections of the report
   c. avoid abbreviations and acronyms unless they are strictly necessary

   Hint: It should be written last, in order to assure it contains the key topics covered in the report.

Here is a good example of a scientific abstract and how it relates to the title:

Title: —Volatile Organic Compounds Determined in Pharmaceutical Products by Full Evaporation Technique and Capillary Gas Chromatography/Ion-Trap Detection‖
Author: Jan Schuberth, National Board of Forensic Medicine, Department of Forensic Chemistry, University Hospital, Sweden
Journal: Anal. Chem., 68 (8), 1317 -1320, 1996

Abstract:
Pharmaceutical products often contain volatile organic compounds (VOCs), which are made up of residual solvents from the manufacturing process and of flavoring additives. These substances may form a "signature" that perhaps could be used to reveal the product source. To study this possibility, a new method for detecting and quantitating VOCs in pharmaceutical preparations is described. It is based on extraction of the dry powder by the full evaporation technique, separation of the VOCs by gas chromatography in a capillary with an apolar stationary phase, and exposure of the compounds by ion-trap detection with the apparatus run in the full-scan mode. The search of some drug substances or pharmaceutical products for VOCs revealed ethanol, acetone, 2-propanol, methyl acetate, toluene, eucalyptol, and menthol, whose concentrations were in the range 0.008-26 mmol/kg of sample. The within-day or between-day precision studies showed, except for methyl acetate, a relative standard deviation less than 13%. The concentrations for the different compounds were at the limit of detection or of quantification in the range 0.4-4.0, respectively, 1-10 mol/kg of sample. Based on the quantitative data, distinct signatures were obtained from synonymous medicines made by four diverse producers. These data indicated that the method provides a means for disclosing the origin of a drug product.

3. **Introduction:** It presents the relevant theoretical background of the project, the applied scientific principles and the proposed solution to the problem. It is based on the relevant information necessary to understand the remainder of the report. A good introduction should address the following questions:

Question	How to address it:
What is the problem?	Describe the problem investigated
Why is it important? What other investigators have done to solve the problem? What remains to be solved?	Summarize what have been done in the past to solve the problem (relevant research). This must include, key terms and concepts, so the reader can understand the merits and significance of the project. State what unanswered question, untested population, untried method are addressed by your research
What is the proposed solution to the problem? What makes your approach unique and/or superior than what others have done?	Explain the merits of your approach in terms of broader impact and intellectual merit State the advantages, and uniqueness of your approach

The introduction must include material from textbooks, laboratory manual, and scientific journals.

Hints:

- Move from general to specific: real problem (world/research) → current literature → your experiment
- Make clear the links between problem and solution, question asked and research design, prior the experiment.
- Be selective, not exhaustive, in choosing studies to cite and amount of detail to include.

4. **Experimental Section:** The experimental section describes the experiment's methods and materials in sufficient detail that another researcher would be able to repeat the experiments and obtain comparable results. It summarizes the entire procedure and clearly delineates the logical progression of the conducted experiment. In the experimental section the writer should:
    - Use complete sentences instead of outlines (Be consistent in voice and tense)
    - Use past tense and passive voice and avoid personal pronouns.
    - Although this section is similar to a recipe for the experiment, it need not be chronological. Describe in detail only the components that are important to the results and conclusions of the report.
    - Assume an audience of experienced experimentalists and provide only necessary information (e.g. type of balance used and its accuracy). Emphasize the conceptual connections among the procedure, equipment, and materials.
    - Include experimental steps, materials, instrumentation, and data collection methods used such as:
        - ***Equipment (Apparatus):*** List only devices of a specialized nature. Provide information about an instrument by giving the manufacturer and model number ***(e.g. "The experiment was done with a Hewlett-Packard Model 5890 Gas Chromatograph equipped with a DB-1 capillary column (30-m x 0.25-mm-i.d. coated with 1 µm of methylsiloxane from J&W Scientific (Folsom, CA)")***. Figures might include the entire experimental apparatus or a detailed view of a unique component critical to the reported results.
        - ***Reagents:*** List and describe the preparation of special reagents only. Do not list reagents normally found in the laboratory and preparations described in standard

handbooks and texts. Provide information about a substance by giving the manufacturer and product purity ***(e.g. Estrone (99% purity, Sigma-Aldrich, St. Louis, MO)).***

- ***Procedure:*** Briefly explain why specific experimental procedures were employed. Since procedures are intended as instructions to permit work to be repeated by others, give adequate details of critical steps ***(e.g. "The solutions used for generating the standard graphs contained, per liter of water, 1% methanol, 10 mmol of ethanol, 2 mmol each of acetone and 2-propanol, and 1 mmol each of methyl acetate, eucalyptol, and l-menthol, or, per liter of methanol, 1.2 mmol of toluene.")***. Diagrams can clarify a complicated experimental setup; use figures when they are more effective than words in describing the experimental technique or apparatus. Always refer the reader's attention to the figure before discussing it in the text. Published procedures should be cited but not described, except where the presentation involves substantial modifications. Very detailed procedures must be presented in the Supporting Information section.
- ***Data Analysis:*** Briefly explain the statistical analysis used to assess and corroborate your findings. Include clear and concise description of the methods and tools employed to interpret the analytical data ***(e.g. "To assay the LOD for the different VOCs, the height of the largest noise peak was measured at the appropriate mass number in a preselected retention time interval. From these data, the peak height equal to 3 times the standard deviation of the gross blank signal,(LOD), was calculated.")***
- ***Safety considerations:*** Describe all safety considerations, including any procedures that are hazardous, any reagents that are toxic, and any procedures requiring special precautions, in enough detail so that workers in the laboratory repeating the experiments can take appropriate safety measures. Procedures and references for the neutralization, deactivation, and ultimate disposal of unusual byproducts should be included.

- A good Experimental section should also address the following:

Question	How to address it:
How did you study the problem?	Briefly explain the general type of scientific procedure you used.
What did you use?	Describe what materials, subjects, equipment (chemicals, experimental animals, apparatus, etc.), and analytical tools you used
How did you proceed?	Explain the steps you took in your experiment. (These may be subheaded by experiment, types of assay, etc.)

5. **Results and Discussion:** contains a comprehensive and coherent summary of the experimental findings, their statistical treatment, and significant results. Combining results and discussion in a single section will give a clearer, more compact presentation. This section requires a strong scientific and technical understanding of experimental and theoretical principles. Therefore the writer should show whether or not he has given any thought to what he accomplished (or learned) in the analysis. Must include applications, implications, principles ***illustrated, improvements with respect to other techniques, and experience gained. In laboratory reports, this is the section where you demonstrate what have been learned from the experience.***

In this section, the writer should:

- State what he has done. Do not include every bit of data generated. Instead, summarize the findings into tables, charts, or graphs which faithfully represent your findings and emphasize the most important results in the light of the experimental objectives.
- Explain findings based on the results you obtained (not on theory).
- Demonstrate the validity and reliability of the results. Draw on existing scientific theory and experiments to elucidate the experimental results.
- Establish the significance and implications of the experimental findings
- Include relevant tables or figures (always refer the reader's attention before showing them) Text and figures should support each other, but also contain enough information to be self-explanatory.

- ***Tables (~3):*** Collect all results and present them as table(s). Report them with their associated standard deviations (if possible). Prepare them in a consistent form, furnish each with an appropriate title, and number (above table), consecutively in the order of reference in the text. Maintain a consistent format for all tables within the report.
- ***Figures (~5):*** Prepare figures in a consistent form, each with an appropriate title, and number (below figure), consecutively in the order of reference in the text. Include any chemical reactions and drawings that are appropriate for the experiment. When preparing a graph consider the following:
  - XY-scatter graphs are best to show trends
  - bar graphs compare magnitudes
  - pie charts show relative portions of a whole
  - plot independent variable on x axis; dependent variable on y axis
  - scale length, width, type, symbols and lines proportionally
  - keep graphs clear and simple
  - use common symbols that are easy to differentiate
  - do not plot more than four or five series on one set of axes
  - clearly label all curves and axis with the parameter being measured (units in parentheses)

- ***Statistical (error) analysis USP<1225>:*** List the source and magnitude of expected errors and their influence upon your results (propagation of error analysis.) Do not go on talking about your own mistakes in this section unless you really know they did affect your results and how.
- ***Accuracy and Reliability of the data USP<1010>:*** Include accepted or literature values if available for all reported quantities, and give the deviations of your experimental values from these quantities.
- ***Nomenclature:*** Should conform to current American usage. Authors should use systematic names similar to those used by the International Union of Pure and Applied Chemistry (IUPAC) and the Chemical Abstracts Service (CAS).

Hints:

- If there is nothing to say about a table or graph included in the results & discussion section, it means that the table or graph should be eliminated from the report, because it provides no information.
- Deciding which data to graph and which to summarize in tables is a skill that will take time to master. Once the key figures and tables are created, they often guide the composition of the remainder of the report.
- A good Results and Discussion section should address the following:

Question	How to address it:
What you have done? How you did it?	Discuss your experimental approach and its advantages to solve the analytical problem.
What did you observe?	Report and discuss the main result(s), supported by representative (most common or best example)
Are the observations valid?	Discuss the figures of merits (validation) of your analysis
What do your observations mean?	Comment on the implications and significance of the most important findings. Describe the patterns, principles, and relationships that the results show.

Question	How to address it:
What are the intellectual merits of your findings?	Explain how your results relate to expectations and to literature cited in the introduction. Do they agree, contradict, or are they exceptions to the rule? Describe what additional research might resolve contradictions or explain exceptions.
What are the broader impacts of your work	Discuss the theoretical implications of your results. Recommend practical applications of your results

6. **Conclusions:** It presents a short summary and assessment of the experiment. The conclusion repeats the key points made in the manuscript. Statements are based on the evidence presented in the report. This section consists of one or two short paragraph(s) reiterating the important results of the experiment, its scientific broader impact and future implications. The main difference between a discussion and a conclusion is that in the former you provide the supporting evidence to justify your hypothesis while the later make statement of the experimental facts that supports or rejects the initial hypothesis. A good Conclusion should address the following:

Question	How to address it:
What do your observations mean?	State the key implications and significance of your findings. Present the patterns, principles, and relationships corroborated by the study
How reliable is your approach?	State the most relevant merits of your approach its advantages and limitations.
What are the intellectual merits of your findings?	Indicate how your analysis agree, contradict, resolve contradictions or represent an exception to the current understanding of a particular phenomenon or technique.
How do your results fit into a broader context?	Suggest the theoretical implications of your results. Suggest practical applications of your results? Extend your findings to other situations and problems. Give the big picture: Do your findings help us understand a broader topic? What are the practical applications of your work?

Note: Research Manuscripts also include a short section that allows the author(s) to thank and acknowledge people, organizations, or financing agencies who added substantially to the work, provided advice or technical assistance, or aided materially by providing equipment or supplies to support the project. The acknowledgements should be short (two or three sentences long) at the end of the report.

7. **References:** Designed to attribute (give credit of) scientific knowledge and ideas to the proper sources. Insert references as endnotes in your document. Sources include textbooks, laboratory manuals, electronic resources and journal articles. Collect citations at the end of the document in the references section, rather than using footnotes at the end of each page. This is standard practice for submitting articles to scientific journals, even if the journal uses a footnote format. The references can be cited in the text using superscript numbers (e.g.,. has been reported by Melendez and coworkers.[2]) There are numerous acceptable formats for references. We will follow the format recommended by the Analytical Chemistry journal of the American Chemical Society. Additional formats and guidelines can be found in the book entitled: ―The ACS Style Guide‖ which can be ordered through any academic bookstore.

   Reference numbers in the text should be superscripted. The accuracy and completeness of the references are the student's responsibility. Use Chemical Abstracts Service Source Index abbreviations for journal names and include publication year, volume, and page number (inclusive pagination is recommended). Include Chemical Abstracts reference for foreign publications that are

not readily available. List submitted articles as —in press‖ only if formally accepted for publication, and give the volume number and year if known. Otherwise use —submitted to‖ or —unpublished work‖ with the name of the place where the work was done and the date. Include name, affiliation, and date for —personal communications‖. These are examples of the reference format:

1. Koile, R. C.; Johnson, D. C. *Anal. Chem.* **1979,** *51*, 741–744.
2. Willard, H. H.; Merritt, L. L., Jr.; Dean, J. A.; Settle, F. A., Jr. *Instrumental Methods of Analysis*. 6th ed.; Van Nostrand: New York, 1981; Chapter 2.

8. **SUPPORTING INFORMATION (20%):** In the interest of a short, more concise and readable report, most peer reviewed journals require authors to report certain types of material in an Appendix called Supporting Information (SI). Such material include:
   a. Detailed data
   b. Equations used to analyze the data that have not already been presented. Do not interpret the data.
   c. Other relevant data, figures, and calculations used in the project.
   d. Data Analysis Summary Outputs
   e. Post lab questions

III. Use the following Information for the Spectrophotometric Analysis of Aspirin to prepare a full Laboratory Report based on the data provided. Remember your report must include all the sections described before.

Spectrophotometric Analysis Of Aspirin

Background:

In this experiment, a sample of commercial aspirin is analyzed. The tablet of aspirin is weighed, a solution of sodium hydroxide is added and heated to obtain the main product $C_7H_4O_3^{2-}$ ion according to reaction (1). The reaction mixture is diluted to 200 mL with distilled water to obtain an initial solution containing the ion $C_7H_4O_3^{2-}$.

$$C_9H_8O_4 \xrightarrow[NaOH]{\Delta} C_7H_4O_3^{2-} + C_2H_3O_2^- \quad (1)$$

incoloro

An aliquot of 1 mL of this initial solution was measured and then diluted to 25 mL by adding a solution of iron (III) chloride, in acid medium, to form the salicylate ion, iron (III) according to the reaction (2). Finally the absorbance of this final solution is measured at the maximum analytical wavelength λ_{max}.

$$C_7H_4O_3^{2-} \xrightarrow[Fe^{3+}(ac)]{H^+} [FeC_7H_4O_3 \cdot 4H_2O]^+ \quad (2)$$

violeta intenso

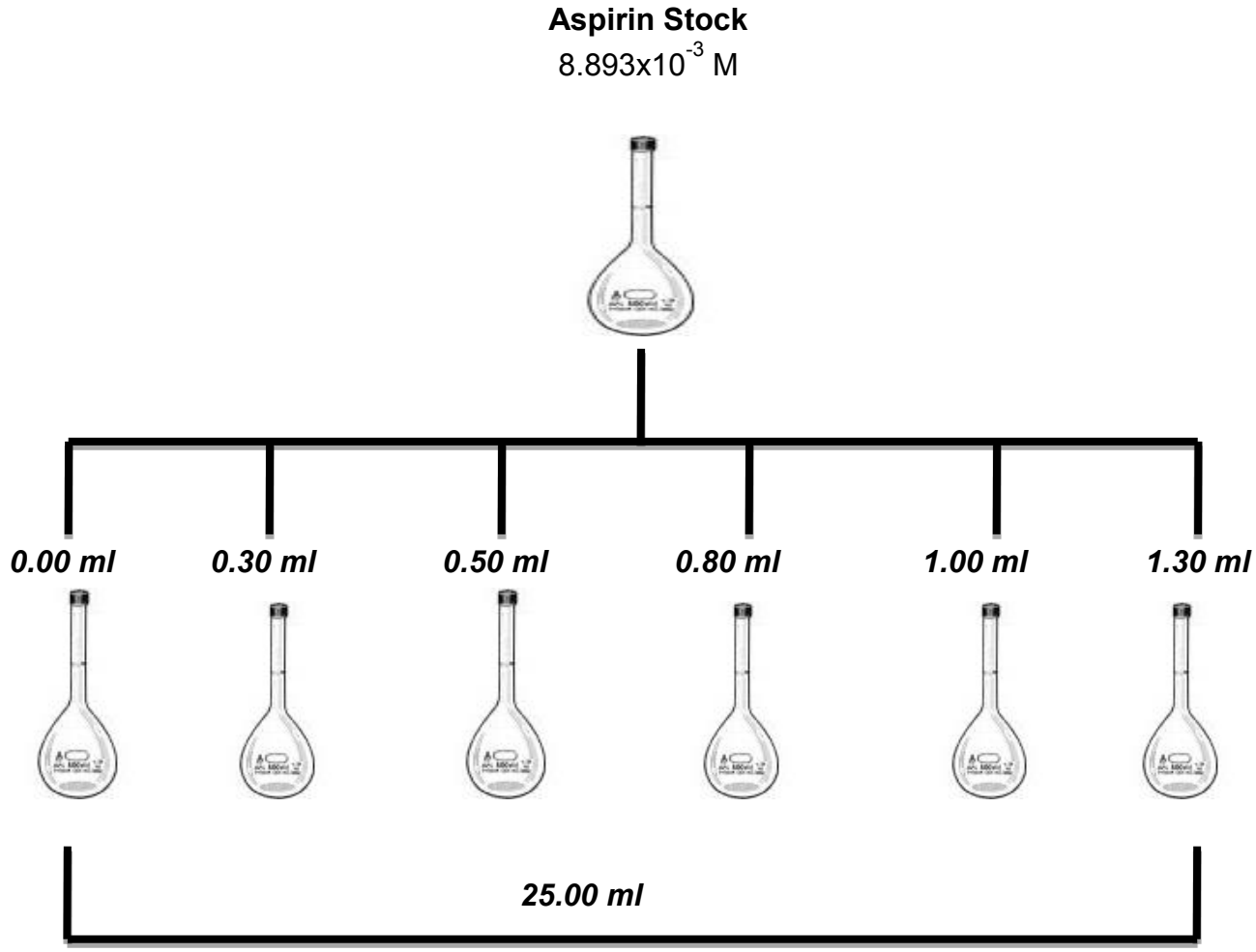
Before the spectrophotometric method can be used, it is necessary to know the wavelength of maximum absorbance of the colored iron (III) salicylate ion. To determine this, the absorbance of a solution containing the ion is used and the absorbance is measured at different wavelengths within the visible region (430- 730 nm). With these data, the graph of the absorption spectrum is prepared and identifies which wavelength maximum absorbance occurs. The spectrophotometer must be set to this wavelength before making absorbance or percent transmittance readings.

In order to investigate the concentration of the sample being analyzed aspirin, prepare a calibration curve or graph of absorbance as a function of concentration of the salicylate ion, iron (III). To this an original or "stock" solution of aspirin of known concentration is provided. Five solutions, whose concentrations can be calculated are prepared by diluting specific volumes of the original solution to a final volume of 25 mL. Finally, the absorbance of each solution is measured. With the absorbance data and concentration of the five solutions, graphics, and linear regression analysis is done

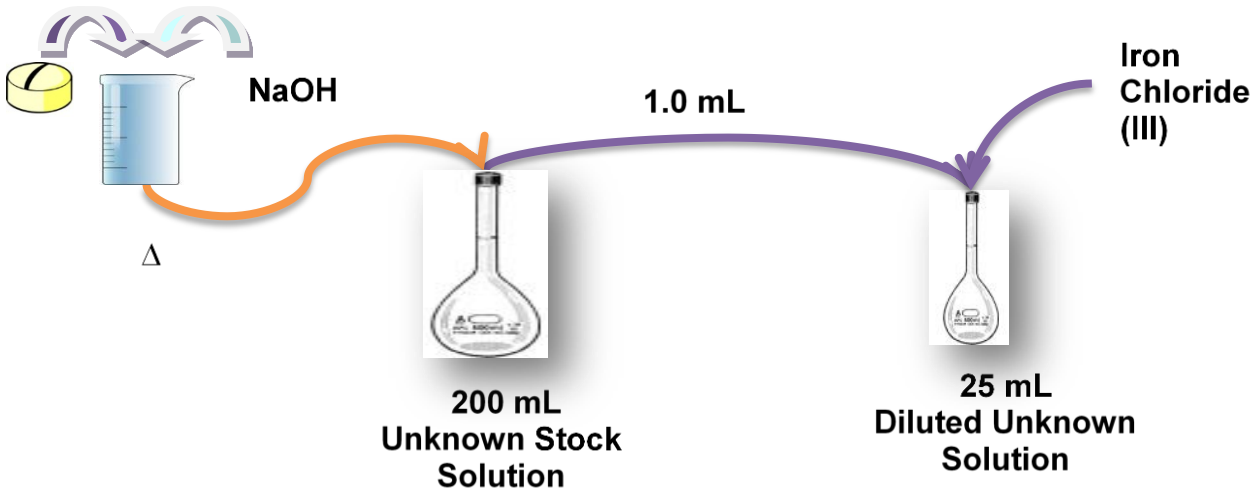
From the above equations we can see that there is a 1: 1 ratio between the reactants and main products. For each mole of Aspirin reacting in the first step produces one mole of $C_7H_4O_3^{2-}$ ion, which in turn produce a mol of the iron (III) salicylate tetrahydrate ion, which is what gives color to the final solution will occur.

II. Procedure

A. Diagram for the preparation of Standard Solutions for the calibration curve plot.



B. Unknown preparation steps diagram:



A. Data:

Aspirin Stock Solution Concentration 8.893×10^{-3} M
Standards Solution volume (V_f) 25.00 mL
Commercial Tablet mass: 0.3281g
Molecular weight of aspirin (acetylsalicylic acid) 180.157 g/mol
Equipment: Beckman DU 640 UV-Vis Spectrophotometer
Plastic Cell

Table #1: Experimental Data for the Spectrophotometric Analysis of iron (III) salicylate tetrahydrate ion

Standard I.D.	Initial Volume (mL)	Absorbance Trial #1	Absorbance Trial #2	Absorbance Trial #3
Blanco	0.00	0.000	0.001	-0.002
1	0.30	0.278	0.28	0.276
2	0.50	0.404	0.407	0.401
3	0.80	0.622	0.595	0.629
4	1.00	0.758	0.755	0.758
5	1.30	0.949	0.951	0.903
Unkown (diluted)		0.626	0.614	0.525

Reference: Delgado,S.; Muñoz Solá, Y.; Solís, L.N., Laboratorio de Química General, McGraw Hill, 2012.

Determine:

- The concentration of each standard solution
- The corrected absorbance for each trial using each blank
- The average corrected absorbance of each solution
- Standard Deviation, %RSD Confidence interval for the corrected absorbance of each solution

Plot and/or calculate

- Calibration curve of **salicylate ion concentration** vs average corrected absorbance
- Slope, intercept, Determination Coefficient (r^2)
- **Aspirin concentration** in the diluted unknown sample, C_{dil}.
- The standard deviation for the diluted unknown concentration, Sc.
- The percentage of aspirin in the tablet (Hint: Consider dilution factors)