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LIST OF EXPERIMENTS

One Week Labs (Wet Chemical Techniques)

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Two Week Labs (Sample Prep and Instrumental Analysis)

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LAB REPORTS

CHEM 311 Lab Reports are submitted as stand alone formal reports (unless otherwise noted) that are to be written in a impersonal voice in typed format. Your lab report should outline the principles of the chemistry and/or instrumentation employed, calibration techniques, data handling, an estimate of experimental uncertainity and a general awareness of the context and significance of the results.

TITLE PAGE AND IDENTIFICATION: Course number. Name of student. Name of partner. Date. Unknown #. The title should provide the reader with both the analyte and the matrix studied and give some indication of the technique employed. E.g., The Analysis of Fluoride Ion in Toothpaste Using An Ion Selective Electrode.

PRINCIPLE OF METHOD: Describe the principles involved in relating the *measured quantity* (e.g., volume of titrant, absorbance, potential etc.) to the *analyte concentration*. For wet chemical techniques, include the stiochiometry of chemical reactions that will be used in the calculation of results. For instrumental techniques, describe the principle of operation of the instrument itself. Diagrams may be useful here for instrumental methods. Do not describe details of the procedure.

PROCEDURE: Not necessary except to specify modifications to the lab manual.

DATA: Tabulate data with descriptive headings and footnotes providing details. Data tables should be able to stand alone providing enough information that the reader could carry out necessary calculations without having to go hunting for additional information. For example, in a data table summarizing titration volumes, be sure to include the titrant concentration and the sample volume.

CALCULATIONS AND RESULTS: Show a representative calculation used to convert measured quantities into reported results. Include calculations used to estimate uncertainities. Figures and graphs must be properly labeled. Use spreadsheets (such as Excel) to carry out repetitive calculations and generate calibration curves (include equation of best fit line and correlation coefficients). In most experiments you will be expected to estimate the experimental uncertainty either as a standard deviation or with a 95% confidence limit.

DISCUSSION: State your result/s and give some context for the magnitude (high, medium or low). For example, report the levels of Fluoride ion in commercial toothpaste, other foodstuffs or drinking water. Be sure to convert to common concentration units, if necessary. Comment on the precision (RSD) and/or accuracy (% bias) of the method using your data and the reported values given in *Standard Methods*. Discuss possible interferents and other sources of error. Conclusion paragraph should clearly report final results for all samples with 95% CL and *n* (# of replicates).

LITERATURE COMPARISON: Briefly summarize one alternative method of analysis used to measure the same analyte. Explain how the analyte is quantified and summarize any advantages/disadvantages of the alternate method. You may use the primary literature such as *Analytical Chemistry* or secondary sources such as *Standard Methods* or an Analytical Chemistry textbook.

REFERENCES: All references cited in the report should be listed as numbered endnotes in the style adopted by *Analytical Chemistry*.

TYPICAL MARKING SCHEME

The following represents a typical marking scheme. Actual marking schemes for a particular lab may vary.

	Mark	Max. Mark
Technique/Preparation – preparedness, work carefully in a clean an organized manner.		**1**
Principle of Method – explain the type of analysis, include relevant chemical equations and/or theory of instrumental operation, including calibration technique. Answers the question: "How is a meaningful quantitative signal generated?"		**3**
Data – complete, clearly presented tables including <u>all pertinent</u> information and uncertainty in measurements.		**3**
Calculations – correct, organized, clearly presented including error analysis to give uncertainty in the final result. Include calibration curves, if any.		**3**
Results – Level of agreement between your result and the known or true value for an unknown or environmental sample.		**3**
Discussion – clearly state your result and give some context for the magnitude (high, medium or low). Comment on the precision (RSD) and/or accuracy (% bias) of method using your data and the reported values given in *Standard Methods*. Discuss possible interferents and other sources of error. Conclusion paragraph should clearly report final results for all samples with 95% CL and n (# of replicates).		**4**
Literature Comparison – include brief overview of essential aspects of an alternate method for the same analyte or alternate analyte using the same method. Use *Standard Methods*, text or library references.		**2**
Layout/Organization – includes pertinent information on title page, proper section headings, labelled figures and/or graphs, all sources of information (references) properly cited as end-notes.		**1**
TOTAL		**20**

		Conc. F (ppm)	Uncertainity (ppm)	Slope
Unknown	Reported			
	True Value			
SRM	Reported			
	True Value			

CHEATING AND PLAGARISM[1]

Cheating and plagiarism are serious offences. There are many forms of beating the system that are considered unacceptable methods of gaining credit. Experience has shown that it is impossible to define every version, and therefore each case tends to be judged separately. The overall aim is to prevent unjustified credit being obtained for work that is not one's own. The penalties for attempting to gain unjustified credit must necessarily appear harsh. It is just as serious for a lab report as for an exam. All lab instructors must refer suspicious situations to the course instructor. The penalties that will be applied include:

- A mark of zero for the work in question
- Referral to the Malaspina University-College Administration, which may include penalties such academic probation or suspension

For disciplinary actions taken by the administration refer to the General Information section of the Malaspina University-College Calendar and visit the website at www.mala.ca/policies.

The notes below give typical chemistry lab examples of situations that may help to clarify the broader definitions given in the Calendar.

- It is unacceptable to
  - record data from samples not prepared by the author without giving due credit to the donor
  - present someone else's data without acknowledging credit (with or without their knowledge)
  - falsify data
  - submit samples not prepared by the author.
  - use ideas or facts from any source without proper reference citation
  - copy another report or portions of a report, be it marked or not
  - copy written material (whether from books, journals, or a website) without using quotation marks. However, keep in mind that direct quotation is not a common practice in scientific writing.
- There is a difference between discussing a lab before work is submitted and producing a collaborative effort. The material submitted for assessment must be the result of the author's individual effort.
- A person supplying material for the purpose of someone else copying or cheating is considered to be equally accountable, and will be subjected to similar penalties.

[1] Adapted from *University of Victoria, Chemistry 235 Laboratory Manual, 2003* with author's permission.

GENERAL LABORATORY PROCEDURES

General Procedures

1. Labs are conducted in pairs. You will need to be organized and divide tasks to complete the labs in the allocated time.

2. Glassware will be provided on an as needed basis during the lab period. Students should come to the lab prepared with a list required glassware and an organized work plan.

Note: You may need to pre-rinse some glassware with deionized water prior to use.

2. In order to avoid contaminating supplied chemical reagents, a sufficient quantity of reagents should be transferred to an appropriate receptacle, e.g. small beaker or weigh boat. A reagent bottle should always be returned to its allocated place after use.

Note: NEVER RETURN A CHEMICAL TO THE REAGENT BOTTLE.

Note: ALWAYS HANDLE PRIMARY STANDARDS AND STOCK SOLUTIONS WITH CARE. CONTAMINATION WILL LEAD TO POOR RESULTS FOR YOU AND OTHERS.

4. At the end of the laboratory period: All glassware should be thoroughly washed (including a final rinse with deionized water) and left on the return cart. All electrical apparatus should be switched off and unplugged. All taps should be turned fully off and all waste should be placed in the appropriate waste container.

5. No student should attempt unauthorised experiments in the laboratory. Students may, on occasion, schedule laboratory work provided permission from an instructor has been obtained. A student <u>must not use</u> the laboratory in the absence of the laboratory supervisor or technician.

6. No chemicals or equipment should be removed from the laboratory at any time.

HOW TO READ AN ANALYTICAL METHOD

1. Preparing Standard Solutions

"make up a series of standards from your stock solution, from X to Y concentration"

There are several important pieces of information hidden in the above instructions.

1. "make up … from your stock solution": all of your standards will originate from this solution. In some cases, where a wide range of concentrations is required, you may need to prepare a 'sub-stock' solution (diluted stock), which you will then use to make your most dilute standards.
2. "a series of standards": in some cases you will be told to make a certain number of standards; in others it is left to your discretion. Generally, four or five standards are used to prepare a calibration curve. You must prepare at least three standards.
3. "standards … from X to Y": this is the concentration range that your standards will cover. Your most dilute standard will have a concentration of X. Your most concentrated standard will have a concentration of Y. Units will depend on the experiment.
4. the dilutions you use to make your standards must be calculated to 'fit' the glassware available. You will have access to 1, 2, 5, 10, 20, 25 and 50 mL volumetric pipettes. You also have 1000, 500, 250, 200, 100, 50 and 25 mL volumetric flasks. Calculate your dilutions so they 'fit' this equipment. (e.g., 1.5 mL into a 150 mL flask is an impossible dilution with your equipment. How else could you get the same dilution factor?)
5. All dilutions used to make standards are made with volumetric glassware.

2. Analytical Shorthand

Rather than spell out exactly how quantities should be measured every time, analytical chemists use a shorthand based on the precise use of language and significant figures. Read through the following examples and 'translate'. If you can't see the difference between the instructions, ask your lab instructor!

"weigh 1 gram of sample"
"weigh exactly 1.0000 gram of sample"
"weigh about 1 g of NaCl exactly"
"add 1 mL of reagent"
"add 1.00 mL of reagent"
"dilute to 1 L in a volumetric flask"
"dilute to 75 mL"

Your translation should include: the type of equipment used, the technique used, and the amount of reagent used.

3. Units

A word on units: you will spend a lot of your time as an analytical chemist converting between units. If you have worked in an analytical lab, you already know about it. Set up a list of conversions for yourself or create an Excel spreadsheet to do this for you. It will save you a lot of time and needless errors later.

mg/L
μg/mL
moles/L
mg/kg
mg/g
wt/wt %
mg/L
ng/mL

Hint: use scientific notation and base SI units.
For example, mg/L = 10^{-3}g/L ≅ 10^{-3} g / 1000 g solution ≡ ‘parts per million’ = 10^{-6} dilution. The symbol ≅ indicates that these quantities are not exactly equal, but are often used that way.

4. The Plan

You will need to have an experimental plan organized prior to arriving in the lab.

1. The information you have in the lab manual must be reprocessed to create an “analytical method”, i.e., a plan. Some of that reprocessing is described above, for example preparing calibration standards.
2. Create your plan using numbered steps or a flow chart, so you can track where you are. Set it up, if you like, so you can check off each step you complete.
3. Number the steps so you can make the most efficient use of your time in the lab.
4. Be aware of time requirements. For example, if your standard will be made from a chemical in the drying oven, it will take some time for it to cool. This might be your first step.

5. “I can’t figure this out …”

Take pity on your Instructor and all the students around you. Ask this question BEFORE your lab period. No, not just 10 minutes before - at least the day before! The more planning you do, the less time you will need in the lab. Planning requires a pen and paper, writing out your sequence of steps, analyzing and modifying as you go. Hint: highlighters are nice, but by the time your whole page is yellow it doesn’t really help you much. Rewrite the methods for yourself in point form in your labbook.

LABORATORY SAFETY

A chemical laboratory is a potentially dangerous environment; the hazards of fire, cuts, burns and poisoning being most prevalent. It is a safe practice to assume all chemical reagents are potentially hazardous. While the use of particularly toxic or carcinogenic reagents is generally avoided, some of the reagents in this lab are dangerous. Check the MSDS and consult your instructor for more information. The first line of defense for skin contact is to flush with plenty of water. Two eyewash stations are provided for the immediate flushing of eye splashes. In the event of an accident, contact your instructor immediately. Safety rules will work only if you obey them and encourage others to obey them. Please familiarise yourself with the following regulations.

1. Personal Safety

- There must be no smoking or eating in the laboratory.
- Students must wear safety glasses at all times. Safety glasses are available. Contact lenses should be removed prior to entering the laboratory. Prescription glasses may be worn.
- Students are recommended to wear laboratory coats in the laboratory.
- Many of the chemicals in the laboratory are poisonous whether taken orally or absorbed through the skin. If any chemical is swallowed the supervisor should be summoned immediately. If any chemical comes into contact with the skin it should be washed off immediately with plenty of water.
- While heating a substance in a test tube, care should be taken to ensure that the mouth of the test tube is not pointing at anyone. A student should never look down into a test tube that is being heated.
- Concentrated acids and bases; strong oxidising and reducing agents; flammable solvents and toxic chemicals should be treated with respect.
- Always wash your hands prior to existing the lab and before eating.

2. Fire

- In the event of fire, the flames should be extinguished with one of the extinguishers in the laboratory and the supervisor notified immediately.

3. Spillages and Fumes

- All breakages and minor spills of chemicals should be reported immediately to the supervisor or technician without delay.
- A receptacle in the laboratory is reserved solely for broken glassware.
- Any experiment involving the evolution of pungent odours or fumes must be carried out in the fume hood.
- Students are accountable for their own actions in the laboratory and this Department will not accept liability for accidents that occur due to irresponsibility on the part of a student or students.

4. Material Safety Data Sheets (MSDS)

- Material Safety Data Sheets summarize physical and chemical properties of all chemical reagents used in this laboratory. In addition, the MSDS sheets contain information on the hazards and toxicity effects. MSDS can be found in lab prep room and should be consulted if there is any question regarding the safety of materials encountered.

MSDS contain information in the following nine categories:

1. product information
2. hazardous ingredients
3. physical data
4. fire and explosion data
5. reactivity data
6. toxicological properties (health effects)
7. preventive measures
8. first aid measures
9. preparation data of MSDS

GENERAL LABORATORY REFERENCES

1. Standard Methods for the Examination of Water and Wastewater (21st Ed.), AWWA, 2005.
2. Water Analysis Handbook, (2nd Ed.), Hach Co., Loveland, 1992.
3. Environmental Sampling and Analysis for Technicians, M. Csuros, Lewis Publishers, Boca Raton, 1994.
4. Drinking Water Chemistry: A Laboratory Manual, B.A. Hauser, Lewis Publishers, Boca Raton, 2001.
5. Laboratory Manual for the Examination of Water, Wastewater and Soil (3rd Ed.), H.H. Rump, Wiley-VCH, New York, 1999.
6. Water Quality and Pond Soil Analysis for Aquaculture, C.E. Boyd, C.S. Tucker, Auburn University, 1992.