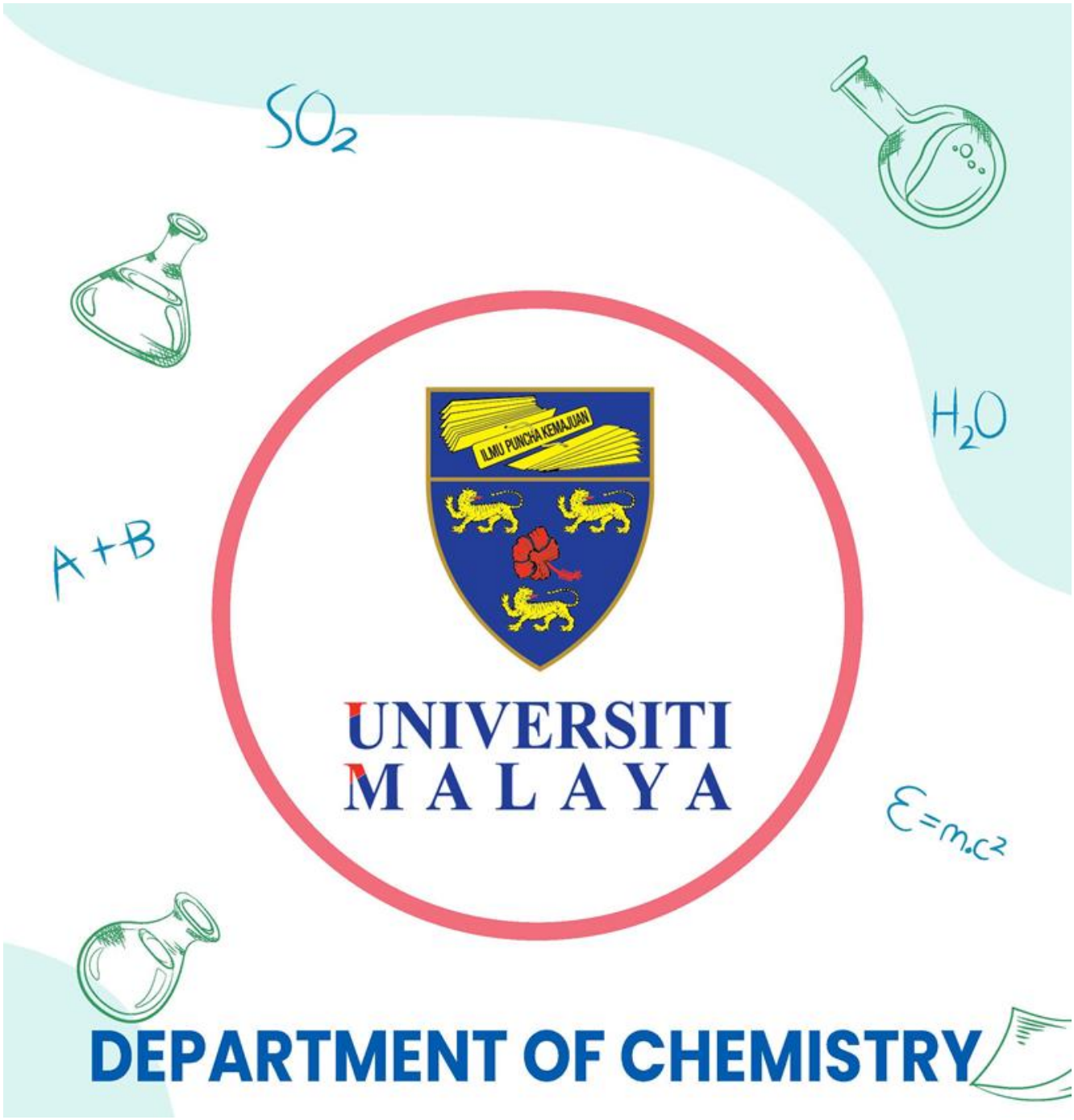


SIC3006 Analytical Chemistry II

SID3006 Advanced Analytical Chemistry

SIC3028 Analytical Chemistry & Instrumentation

SIC3043 Advanced Analytical Chemistry

Laboratory Report Writing

(From: William, I., 2001.Environmental Chemistry: A modular approach, West Sussex, John Wiley & Soms, Ltd)

1. Introduction

As a student, you will be required to submit essays, laboratory and project reports to your lecturers for assessment. In future years, as a researcher, technician, teacher, academic, industrialist, civil servant, media correspondent, author, salesperson or politician, you may be required to write a range of scientific text targeted at a specific audience. This prospect may terrify you; many people regard writing as difficult, and something to be delayed or avoided. In fact, scientific writing is a skill, which, like tying your shoelaces or performing titration, can mastered with practice and perseverance. Like any other skill, scientific writing can be developed into something that will give you confidence, satisfaction and pleasure.

At undergraduate level, laboratory reports are very important components of assessed work, and consequently, it is worth trying to produce good quality reports. As chemist, laboratory reports, are written for several reasons. One reason is to communicate the laboratory work to management. In such situations, management often bases company decisions on the results of the report. Another reason to write laboratory reports is to archive the work so that the work will not have to be done in the future. Laboratory reports are intended to demonstrate some or all of the following:

- you have performed and understood an experiment;
- you have some knowledge of the theoretical basis of the experiment;
- you can process/interpret the data obtained from an experiment;
- you can relate fundamental or derived laws to the outcome of the experiment;
- you can present these ideas/results in an appropriate context and can evaluate their significance.

2. Effective Scientific Writing

1. Remember the purpose of your writing – communicate clearly, concisely and accurately.
2. Consider your audience (tutor/lecturer) and the assessment criteria.
3. Use appropriate format.
4. Plan and arrange your ideas in a logical order.
5. Treat what you write first as draft.
6. Make sure your grammar, spelling and punctuation are correct.
7. Ensure the first draft is clear enough.
8. Re-read and edit your first draft as necessary.
9. Proof-read the final draft, correcting any remaining mistake.

3. Grammar and Style

All the text in your report should be grammatically correct, properly punctuated and comprise complete sentences. The overwhelming majority of scientific reports are written using the impersonal Third Person / Past Simple Tense / Passive Voice form, acoiding, if possiblem the use of the personal pronounce (I, we, or you). The following examples illustrate what is intended:

- Preferred “The samples were stored at 0 °C”
- Not preferred “I stored the samples at 0 °C”

4. Presentation

Laboratory reports should be good to look at; a well-presented report will please the reader, give him/her confidence in the report and will aid assessment. A cover page will aid the presentation of your work, as well as providing important information to your assessor. The cover page should have (Figure 1):

- Course title and code;
- Number of experiment;
- Your report title;
- Your name and matric number;
- Name of your group members
- Date of submission;
- Name of Lecturer / Tutor.

SIC3006 Analytical Chemistry II

Experiment 1

ANION ANALYSIS USING ION CHROMATOGRAPHY

Name: _____

Matric No:_____

Group member: (1) _____ (2) _____

Date of Submission: _____

Lecturer: _____

Tutor: _____

Figure 1: Example of cover page.

Laboratory reports should always use SI units. Unit is very important for all measurement. Without units much of our work as scientists would be meaningless. We need to express our thoughts clearly and units give meaning to the numbers we calculate. Knowing the units of measurement that correspond with a number can give you so much more information than a digit sitting there by itself. Units can:

- Help to show another person the exact amount you have;
- Assist in solving a mathematical problem, especially in chemistry, where you can follow the units to get to the answer;
- Show which measurement system the person is using (i.e. metric or standard).

Proper pagination of your reports will assist you to structure your work, as well as being good practice. It will also assist the reader / assessor to 'navigate' your report, thus making it easier to find relevant sub-sections, table, figures, etc. Pages containing preliminary information (e.g. cover page) are paginated in small Roman numerals (I, ii, iii, etc.), whereas pages of the main body of the report are given in Arabic numerals (1, 2, 3, etc).

5. Structure of the Laboratory Report

Basic structure for laboratory reports:

- Cover page (refer to section 4)
- Aims / Objectives of the Experiment
- Introduction
- Materials and Methods (Experimental)
- Results
- Discussion
- Conclusions
- References
- Appendices (if related)

5.1 Aims / Objectives of the Experiment

The aims or objectives of the experiment should clearly and briefly state the purpose of undertaking experiment. They usually include specific overall aims of the experiment. For example, in Experiment 1 that determine the concentration of anions in different types of tea leaves.

- To determine the concentration of anions in different types of tea leaves using ion chromatography.

You should always refer back to your aims in the Conclusions section of your report and comment upon whether they have been achieved satisfactory.

5.2 Introduction

The introduction should establish the context of the experiment, and explain the rationale for undertaking it (i.e. why is it worth doing at all). Here, you should provide some background information on the problem under investigation, such as the source of the pollutant under investigation and any potential health/environmental effects. This section can also involve a iv description of the theory relating to the experiment and the experimental technique(s) to be used. It should leave the reader with the feeling that the report has a general relevance and that to read on would be worthwhile.

5.3 Materials and methods

This section should contain a concise but adequate description of all of your experimental materials and procedures so that your results could be verified independently. Materials, too should be as fully described as is necessary for replication. The details of the apparatus / instrument (e.g. UV-Vis Spectrophotometer; GC-FID, AAS, etc) used should be included at this section. There is also no need to repeat routine instructions for using apparatus or equipment where they are well-known or available in manufacturers' instruction. Figure 2 shows the example of the description for chemicals and instruments.

Any form of sampling procedures must be very fully described – both the sampling techniques and the sampling strategy. Sampling usually undertaken to obtain some estimate relating to a population. Similarly, locations and study areas should be described well enough for a reader to duplicate , locate or visualize.

2.1. Chemicals

Parabens (esters of 4-hydroxybenzoic acid, MeP, EtP, PrP, BuP and BzP), phenol and nitrobenzene (NB) were obtained from Fluka. *Tert*-butanol (*t*-BuOH) was obtained from Sigma–Aldrich. All solvents (Merck) were of the HPLC grade. Individual parabens stock solutions were dissolved in boiled ultrapure deionized water (Elga, USA). A mixture of BSTFA (*N,O*-bis(trimethylsilyl)trifluoroacetamide) and TMSCl (trimethylchlorosilane) in a ratio of 99:1 was obtained from Supelco (USA). Sodium phosphate monobasic and Sodium phosphate dibasic were purchased from Sigma and Riedel-de-Haën, respectively.

2.5. Instrumental

All HPLC analyses were performed using Shimadzu HPLC system consisted of a LC-20AT pump, a SPD-M20A diode array detector, a SIL-20AHT auto sampler, a CTO-20AC column oven and a CBM-20A communication bus module (Shimadzu, Japan). A reversed-phase Chromolith RP-18 monolithic column (100 mm × 4.6 mm; Merck, Germany) was used for separation.

Analysis of degradation by-products was carried out using a Hewlett–Packard Model 6890 GC, with a HP-5 (5% phenylmethylpolysiloxane) column. The detail of the setting and the GC temperature program was given in previous study (Tay et al., 2009).

Figure 2: Example of writing the description for chemicals and instruments

5.4 Reporting Results

Clearly, the Results are an exceptionally important part of your report and great care should be taken in their presentation. Over the years, a number of conventions have developed in the reporting of results. It is important to open your Results section with appropriate text rather than by just presenting tables of data. A table must follow, and never precede, the frist reference to it in the text. You should not leave it to the reader to interpret tables – that is your job. An acceptable format is of the type, 'The data presented in Table 1 show that ……'. Indeed, the reader should be able to appreciate the significance of the result without reference to any talbe of data; the data are evidence to support your statements. While tables are used to present the data, figures can be helpful in interpreting them.

Tables

Tables are the main vehicles for conveying data to the reader. A table can be considered as a complete entity, in a sense, should be able to exist separately in the text. A well-constructed table does not need a lengthy explanation on how it is to be interpreted but should be selfexplanatory and be characterized by its simplicity and unity. The caption (on top of the table) v is clearly important if the table is to stand as a separate entity. Table 3 is a well laid out and clear example.

Table 3. Characterization of the leachate collected from the Gramacho Metropolitan Landfill used in this work (n = 4 samples).

Parameters	*Average value*	*Standard deviation*
pH	8.3	0.3
Total alkalinity (mg $CaCO_3$ L^{-1})	8857	1480
Carbonate alkalinity (mg $CaCO_3$ L^{-1})	450	490
Bicarbonate alkalinity (mg $CaCO_3$ L^{-1})	8374	1917
Total ammonia nitrogen (mg [N-NH_3] L^{-1})	1998	387
Chloride (mg L^{-1})	3196	862
Dissolved Reactive Phosphorus (mg L^{-1})	7.5	1.3
Total Solids (mg L^{-1})	9390	2087
Total Suspended Solids (mg L^{-1})	53	31
DOC – dissolved organic carbon (mg L^{-1})	935	71
COD chemical oxygen demand (mg L^{-1})	3332	523
BOD - biochemical oxygen demand (mg L^{-1})	141	45

Figures / Graphs

Laboratories exercises will oftern involve the production of graphs from the data collected. A graph can provide much more information than a set of data. It gives a visual representation of trends and relationships, and permits the prediction of what happens between the known points. Graphs are commonly labeled as Figure in lab reports. As tables, appropriate captions (or titles) should be added at the bottom of the graph (refer to Fig. 3). Well-drawn graphs can greatly enhance the effectiveness of display and interpretation of the results presented in a report.

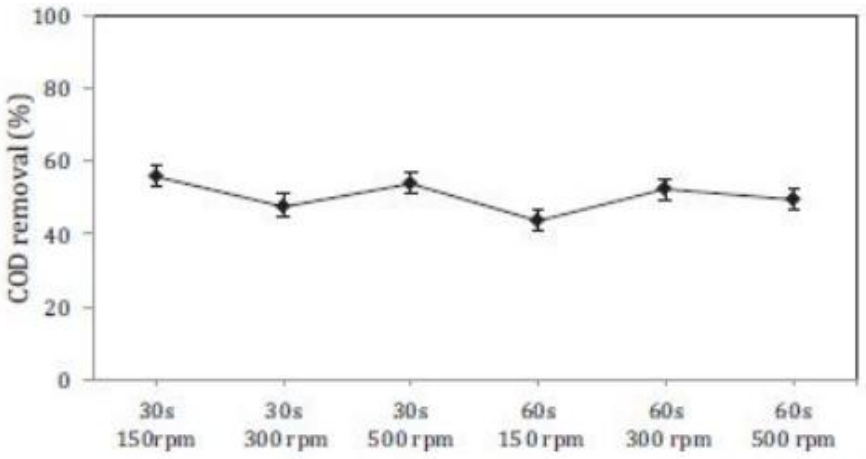


Fig. 3. Removal of COD for various mixing times and stirring speeds (experimental conditions: pH = 4.0, $FeCl_3$ dosage = 1400 mg L^{-1} and temperature = 23°C).

5.5 Discussion

The Discussion should draw all the threads of the report together and is, arguably, the most important part of the report. The discussion offers the widest scope for individual freedom of expression, and may include items such as the following:

- A comparison of the results with those obtained or published elsewhere;
- A discussion of the significance of the data in an appropriate context;
- Comments on the value of the results in a wider scientific, environmental or even commercial context.
- A discussion of the possible limitations of the methods; vi
- Comments upon the precision, reproducibility or repeatability) of the results, as well as on the accuracy, if known.
- A discussion of effectiveness and limitations of the experiment and any statistical treatment of the data. Attention should be drawn to any fault/problems with the chemicals or equipment used and to any deficiency in the assumptions upon which the experiment is based. Modifications and improvements should be included if appropriate.

5.6 Conclusions

The Conclusion section should summarize the main findings of the experiment. It is not a summary of your work programme or a description of the research carried out. It is often helpful to use ‘bullt points’, each no more than two or there lines, to summarize your results. This enables you, lecturer and tutor to see, at glance, whether you have addressed all of the important areas and helps you to check that you have covered everything that you wanted to and listed in the objectives.

5.7 References

Citing references

References may be cited in the text in a number of ways, depending upon your style of writing or the context of your reference. However, there are convention that should be followed, as shown below – note the use of brackets.

- Natural levels of carbon monoxide are low, typically in the range 20 – 200 ppb (Grimes and Clement, 1993).
- Kinnear (1998) describes a system for sampling PM10 on an hourly basis, while Hegarty et al. (2001) describe a system for the continuous sampling of PM10. [Note: “Hegarty, Scanlon and Chan (2001) is written as Hegarty et al. (2011)]

If reference has two authors or less, the family name of all author(s) should be mentioned in the reports. If a reference has more than two authors, only the first is mentioned with “et al.” “et al. translates as “and others”.

You may want to cite an official or company report, or government paper, where there is no specified author or the authorship belongs to a committee. In such cases, you normally cite the body responsible for publishing the paper or report. Thus, in the text, the body responsible for publishing the paper is cited with the year of publication, e.g. (EvironTech Ltd, 2000).

The Reference Section

The Reference section must include details of all references that have been cited in text. It does not include peripheral reading. The details of each reference include the following: name(s) of the author(s) (surname first, with a comma), the year of publication, and the title of the publication. In the case of books and reports, the name of the publisher and place of publication is also given, There is more than one way of presenting this information; the following example illustrate the use of upper and lower case letters, italics, punctuation marks and general layout.

Books

Example: Roberts, M.B.V. (1984). Biology: A Functional Approach (3rd Edn). Nelson Publishers, London.

Book Chapters

XYZ, F.M. (Year Published). Title of chapter In F.M. XYZ Editor (Ed.), Title of book/anthology (pp. Pages). Publisher City, State: Publisher.

Article in Journals

XYZ, F. M., & ABC, F. M. (Year Published). Article title. Journal Name, Volume (Issue), Pages.

Websites

Satalkar, B. (2010, July 15). Water aerobics. Retrieved from http://www.buzzle.com

6. PLAGIARISM

Plagiarism is the representation of another person's published or unpublished work or ideas as your own by using an extensive unacknowledged quotation. In academia, plagiarism carries heavy penalties; your mark for any assessd work may be significantly reduced and you may be open to accusations of academic misconduct. However, this does not mean that all of your work must be completely original; expressing views that are influenced by other authors is a consequence of shared knowledge and reflection of wide reading. In order to avoid accusations of plagiarism, you should clearly reference sources by using the conventions outlined above

Laboratory Report Marking Scheme

Section 1: Lab Performance (Total 20%)

1. Pre-entering lab (5%)

Score	Criteria
0	No preparation of experimental procedure, no proper attire-shoes; goggle; lab coat.
1-2	Summary of procedures too brief, lack of details and confusing; incomplete safety attire.
3-5	Presents easy to follow steps in lab experimental, logical and adequately detailed; safety attire checked.

2. Skill & Techniques (15%)

Score	Criteria
0	No skill is demonstrated.
1-5	Wrong glassware used, wrong technique, spillage and wasting of chemicals.
6-10	Right glassware used, incorrect or lack of lab technique.
10-15	Presents correct lab skill, clean and tidy.

Section 2: Lab report (Total 60%)

Section	Total Mark	Rubric	
Title	5	0-1	• No title, or • Too brief (e.g. “Lab report”; “Mercury in fish”; Ascobic acid in fruits”, etc).
		2-3	• Too long, or • Does not identify the complete subject of study (E.g “Determination of mercury”; “Determination of lead”, etc).
		4-5	• Identify the complete subject of study and encapsulates the purpose of the report/study.
Objective	15	0	• Section missing completely.
		1 - 7	• Be too vague, ambitious or broad in scope. • Just repeat each other in different terms. • Just be a list of things related to the topic. • Contradict with methods. • Does not identify subject of study.
		8 - 15	• Concise and brief. • Be interrelated and describes how to achieve that objective. • Clearly identify the subject of study. • Related to the experiment that has been done.
Introduction	10	0	• Section missing completely.
		1 - 5	• Background info only from lab manual.
		11 -15	• Clearly written, well structured, with evidence of extra reading. • Clear outline of study’s hypotheses. • Does show something novel in it as compared to the supplied handout/laboratory manual.

			• Does include the rationale for performing the experiment.
Experimental	10	0	• Section missing completely.
		1 - 5	• One or more subsections (e.g. chemicals or instrumentation) are missing. • Confusing statement. • Parts have been included under the wrong sub-section.
		6 - 10	• Contains all of the relevant information about the method used; clearly and systematically described in such a way that a reader could replicate the study from the description.
Results	20	0 – 5	• Graphs or tables are included without caption and any written explanation, • Has some writing without tables and graphs. • Very poor presentation of the collected data.
		6 – 10	• Has included the raw data in tables • Poor presentation of data (e.g. no graphs; wrong graphs; irrelevant graphs, no label and caption) • Inaccurate explanations.
		11 – 15	• Has presented the data in a logical format (e.g. graphs, tables) • Show some understanding with explanation, but some relevant information has been omitted • Graphs and tables are not label accordingly/correctly. • Standard deviations or standard errors missing from the presented data (if related).
		16 – 20	• Logical sequence, • Clear presentation with relevant and clear explanation, • Figures and tables are well labeled.
Discussion	20	0 – 5	• No attempt to relate results to relevant theoretical and empirical research • Does not understand of what the study was about.
		6 – 10	• Poor structure, wrong order, shows little understanding of the experiment.
		11 – 15	• Poor structure, but contains essential elements, or • Good structure with some missing elements.
		16 – 20	• Well organized and clearly written • Clearly summarize the obtained results • Does show attempt to relate the findings to previous research • Does show ability to evaluate the weakness and limitations of the study • Does include sensible suggestion for possible improvement.
Safety caution	5	0-1	• Section is not presented
		1-3	• Sentences are not in complete, focusing on minor or lack important steps.
		4-5	• Tabulated at least 3 major and most important safety caution.
Conclusions	10	0	• Section missing completely
		1 – 5	• Conclusion is drawn but not supported by experimental evidence. • No sensible conclusion is drawn.

			• No clear evidence of a thorough understanding of the experiment and/or theory behind the experiment.
		6 – 10	• Conclusion is drawn and supported by experimental evidence. • Sensible conclusion is drawn. • Shows clear evidence of a thorough understanding of the experiment and/or theory behind the experiment.
References	5	0	• Reference not included in the report
		1 - 3	• Incomplete references to the books or any other sources used in report.
		4 - 5	• References in the text and in the reference list conform in all respects to the formatting convention (e.g. APA format) • Complete references to the books or any other sources used in report. • References in text are matched with references in reference list (e.g. no missing references)
Total Mark	100		

Section 3 Assessment of Understanding/Revision on conducted experiments (20%)

**For Section 3 Assessment-it is up to the lecturer in-charge to decide whether want to carry out a simple test or not. If choose not to, the 20% marks will be allocated back to Section 2- Lab report*

Score	**Criteria**
0	Unable to answer any questions.
1-5	Very little attempt to answer question correctly.
6-10	Most answers are incorrect, and some are irrelevant to the question type.
11-15	Some answers maybe very short or incomplete.
16-20	Questions are answered to the best of abilities and answers match the question types.

Late Report: -1 marks / day

Analytical Chemistry II (Mini project)

No.	Title	Descriptions	Objectives
1	ANION ANALYSIS USING ION CHROMATOGRAPHY	Quantitative experiment for the determination of anion in different tea leaves using ion chromatography (IC). In this experiment, students will be exposed to the application of ion chromatography (IC) in analyzing anion contents in different types of tea leaves. The goals of this experiments are to be familiar with 1) stock and calibration standard preparation, 2) sample preparation, 3) IC application, 4) sample analysis & data interpretations, and 5) report writing.	1. To identify the best methods for anion analysis in tea leaves using ion chromatography. 2. To develop practical skill in stock & calibration standards preparation and sample preparations. 3. To gain basic knowledge in instrumentation handling from sample analysis to data interpretation 4. To determine the concentration of anions in different types of tea leaves
2	ANALYSIS OF BISPHENOL-A (BPA) IN THERMAL PAPER USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)	Quantitative experiment for the determination of Bisphenol-A (BPA) in thermal paper using High Performance Liquid Chromatography (HPLC). The goals of this experiments are to be familiar with 1) stock and calibration standard preparation, 2) sample preparation, 3) HPLC application, 4) sample analysis & data interpretations, and 5) report writing.	1. To identify the best methods for BPA analysis in thermal paper using HPLC 2. To develop practical skill in stock & calibration standards preparation and sample preparations. 3. To gain basic knowledge in instrumentation handling from sample analysis to data interpretations. 4. To determine the concentration of BPA in different types of thermal paper.
3	ANALYSIS OF TRACE METALS IN TEA LEAVES BY FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY (FAAS)	This experiment is designed to acquaint in student with the techniques of FAAS for the analysis of metals. The use of an FAAS is illustrated for the determination of metals in different types of tea leaves and/or tea leaves products. The goals of this experiments are to be familiar with 1) stock and calibration standard preparation, 2) sample preparation, 3) HPLC application, 4) sample analysis & data interpretations, and 5) report writing.	1. To identify the best methods for metal analysis in tea leaves/milk using FAAS. 2. To develop practical skill in stock & calibration standards preparation and sample preparations. 3. To learn about digestion techniques as follows: i. Hot plate digestion ii. Microwave digestions 4. To gain basic knowledge in instrumentation handling from sample analysis to data interpretations. 5. To determine the concentration of metals in different types of samples.

4	ANALYSIS OF TRACE METALS IN MILK BY FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY (FAAS)	Milk is a good source of calcium (Ca), magnesium (Mg), phosphorus (P), potassium (K), selenium (Se), and zinc (Zn). This experiment is designed to acquaint in student with the techniques of FAAS for the analysis of metals. The use of an FAAS is illustrated for the determination of the Ca, Mg, Se and Zn in all types of milk and/or milk products.	6. At the end of this experiment, students should be able to apply FAAS for the analysis of trace/heavy metals. *types of samples can be changed accordingly.
5	AN INTRODUCTION TO CYCLIC VOLTAMMETRY	The goal of this experiment is to become familiar with using a modern electrochemical potentiostat, to determine the concentration of potassium ferricyanide(III), 99%, K3Fe(CN)6, in an unknown solution, and to measure the diffusion coefficient for the ferricyanide anion, [Fe(CN)6]3-/4-. This procedure illustrates how the current observed in a cyclic voltammetry experiment depends upon experimental parameters such as concentration and sweep rate.	1. To study the effect of concentration and sweep rate on the peak height and peak potential. 2. To calculate the diffusion coefficient for the ferricyanide anion based on the slope of the calibration plot generated. 3. To determine the concentration of unknown solution using the equation generated from the calibration plot.

Experiment 1

ANION ANALYSIS USING ION CHROMATOGRAPHY

1. Synopsis

In this experiment, students will be exposed to the application of ion chromatography (IC) in analyzing anion contents in different types of tea leaves.

The goals of this experiments are to be familiar with 1) stock and calibration standard preparation, 2) sample preparation, 3) IC application and 4) sample analysis & data interpretations.

2. Objectives

a) To identify the best methods for anion analysis in tea leaves using ion chromatography.
b) To develop practical skill in stock & calibration standards preparation and sample preparations.
c) To gain basic knowledge in instrumentation handling from sample analysis to data interpretation
d) To determine the concentration of anions in different types of tea leaves

3. Experimental Apparatus

- 250mL beaker
- Hot plate
- Glass cover/ watch glass
- Label
- 0.45 um filter paper
- Sterile Plastic/sample bottles
- 100mL volumetric flask (stock solution 1000ppm & 100ppm)
- 25mL VF (standard solution), 5 units
- Micropipette

4. Reagents and Chemicals

- Sodium sulphate, Na_2SO_4
- Sodium phosphate, Na_3PO_4
- Sodium nitrate, $NaNO_3$
- Sodium chloride, NaCl

5. Sample

- Tea leaves

6. Procedure

All glassware used for electrochemistry should be as clean as possible. The solvents and reagents used to make solutions should be as pure as possible. It is a good idea to use deionized, ultrafiltered (DIUF) water or "conductivity water" or "HPLC grade water" for the final rinsing of glassware and for all solution preparation.

A. SOLUTION PREPARATION

1) **1000ppm anion stock solution (100 mL)**

***** These stock solutions should be provided to a group of 3-4 students:
[NaCl formula mass is 58.44 g mol^{-1}; $NaNO_3$ formula mass is 84.99 g mol^{-1}, Na_3PO_4 formula mass is 163.94 g mol^{-1}; Na_2SO_4 formula mass is 142.04 g mol^{-1}; Molar mass of Na is 22.99 g mol^{-1}]

The anion stock solution can be prepared by dissolving _____ grams of NaCl, _____ grams of $NaNO_3$, _____ grams of Na_3PO_4, and _____ grams of Na_2SO_4, in ultrapure water to obtain a 100 mL final volume.
** show full calculations in your report*

2) **Calibration standard solution (25 mL)**

Each group of students should prepare five standard solutions with various concentrations of anion stock solution ranging from 0 to 25ppm. The solutions can be prepared by pipetting various volumes of the stock solution into a series of five 25 mL volumetric flasks. Fill up the concentrations data in the table below:

Standard concentration (ppm)	Flask volume (mL)	Pipette volume (mL)
	25	
	25	
	25	
	25	
	25	

B. SAMPLE PREPARATION

1) Grind tea samples to powder form
2) Put 2g of grinded tea into a 250mL beaker and add 100ml of ultrapure water. Boil at 100°C (cover with watch glass).
3) Once the water starts to boil vigorously, leave the sample for 10 minutes (infusion time) for complete extraction of inorganic anion. Then, keep the sample solution to cool down at room temperature.
4) Filter the sample and stored in a sample bottle. Samples prepared in triplicates.

C. ELUENT PREPARATION FOR SAMPLE ANALYSIS USING IC

1) Eluent must be prepared as follows:

Sodium bicarbonate	5 ml
Sodium carbonate	40 ml
in 500 ml/ 1000mL	volumetric flask

2) Fill the eluent in the designated bottle **(Figure 6.1)**

Figure 6.1

D. SETTING UP THE SOFTWARE

1) Click on the **Chromeleon icon**  on the computer desktop
2) Click on the **Start** button and select **All Programs > Chromeleon > Chromeleon.**
3) To display the Dionex ICS-1100 Control panel, click the tab labeled with **Dionex ICS-1100 Timebase (Figure 6.2)**.

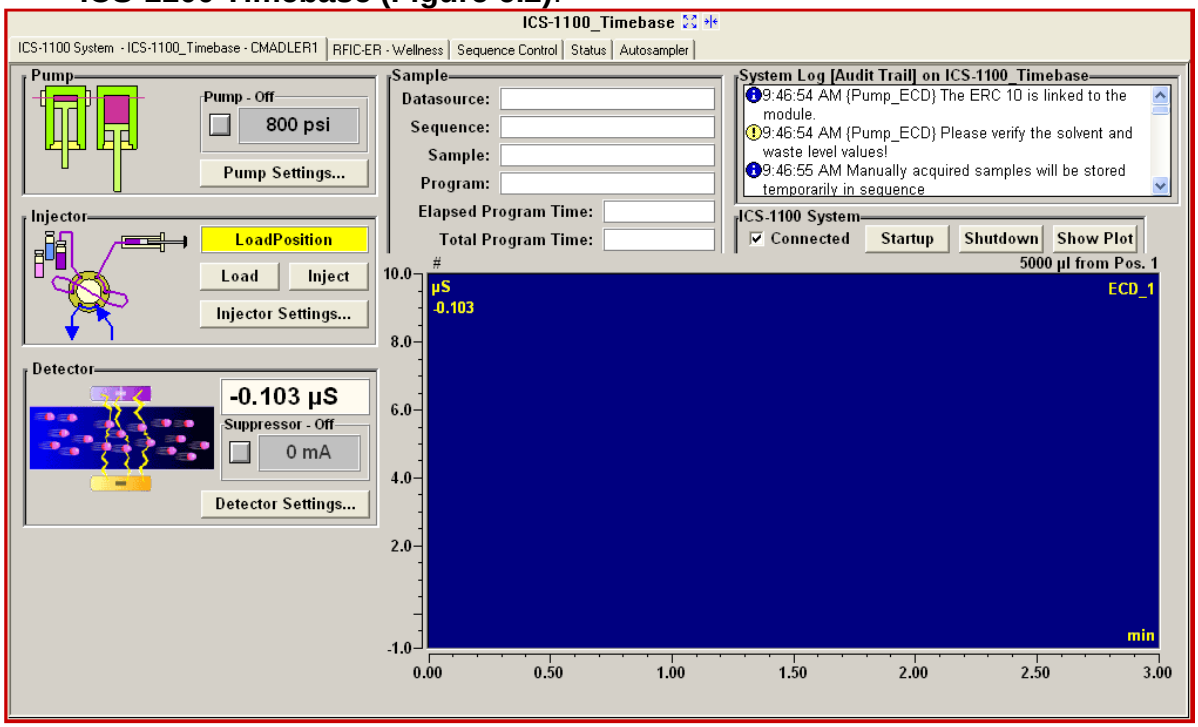


Figure 6.2

4) Go to **Pump Setting** and update on the eluent bottle. Pump Setting configuration are as stated in the **Figure 6.3** below.

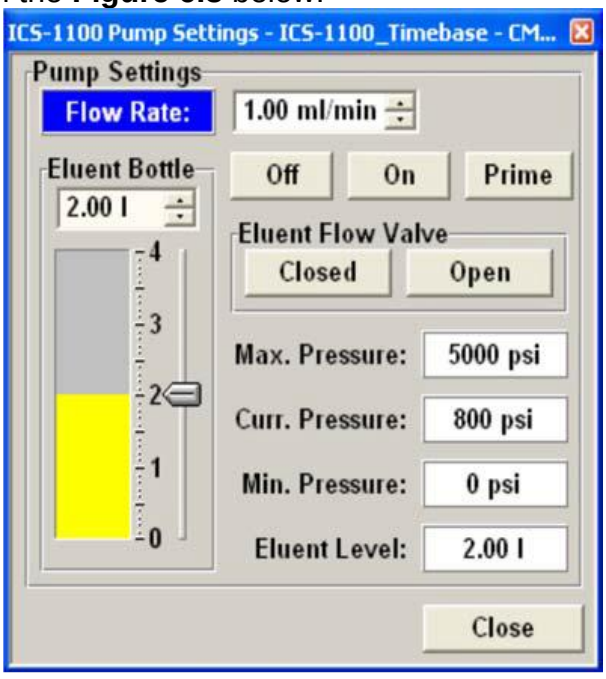


Figure 6.3

5) Create a new file.

- To create a file, Click on **Sequence**, go to **File** and **Save as**. Fill a name file, then click **Save**
- Right click to append sample (i.e to add new sample)
- The status of all samples that are not run should be changed to **'FINISHED'** except for the first row, which is UPW (ultrapure water) that is used as Blank. Then, click **Save.**
- Go to **Programme** and change the time to the desired analysis time. Then, **Save.**
- The programme for UPW should be switched to AS14A. Then, **Save.**
- Click on **UMqnt** and change the standard concentrations according to your preparation. Then **Save.**
  ** Note that the sample list should always start with 1) UPW (blank), and followed by 2) 5 calibration standard and 3) Samples*

E. SAMPLE RUN

- Click on **Control** drag to **Acquisition Off**, select **Yes** and inject deionized water into the system **2-3 times**.
- Make sure that the status of the **UPW** is on **SINGLE**, not **FINISHED**. Click on **Sequence** drag to **Batch,** select **Start** and press **OK**. Check the system panel and observe the chart of deionized water.
- Once the chart of deionized water sample obtained baseline, it's an indication that the system is ready for analysis.
- *Change the status of **Standard 1** from **FINISHED** to **SINGLE**. **Save** and inject **Standard 1**. Click on **Sequence** drag to **Batch,** select **Start** and press **OK**. Run the standard and observe its chart until the end of its programmed analysis time. Make sure the status of the batch (**Standard 1**) changes to **FINISHED** before you go to the next sample/standard.
- Repeat the above step * for the remaining standards and samples.
- Navigate around the **Sequence** icon to obtain the concentrations, calibration curve, area and standard deviation.

References

1. Cummins, P. M., Rochfort, K. D., & Connor, B. F. (2016). Ion-exchange chromatography: Basic principles and application. Protein Chromatography, 209-223. DOI:10.1007/978-1-4939-6412-3_11
2. Frenzel, W., & Michalski, R. (2016). Sample Preparation Techniques for Ion Chromatography. Application of IC-MS and IC-ICP-MS in Environmental Research, 210–266. doi:10.1002/9781119085362.ch8
3. Minca, I., Josceanu, A.M., Isopescu, R.D., Guran, C. (2013). Determination of Ionic Species in Tea Infusions by Ion Chromatography. U.P.B. Sci. Bull., Series B, Vol. 75, Issue 3.
4. Thermo Fisher Scientific Inc. (2012). Dionex ICS-1100 Ion Chromatography System Operator's Manual

Experiment 2

DETEMINATION OF BISPHENOL-A (BPA) USING HIGH PERFORMANCE LIQUID CHROMATOGRAPHY (HPLC)

1. Synopsis

For this experiments, determination of Bisphenol-A in thermal paper using HPLC will be conducted.

The goals of this experiments are to be familiar with 1) stock and calibration standard preparation, 2) sample preparation, 3) HPLC application & handling and 4) sample analysis & data interpretations.

2. Objectives

1. To identify the best methods for BPA analysis in thermal paper using HPLC
2. To develop practical skill in stock & calibration standards preparation and sample preparations.
3. To gain basic knowledge in HPLC application and handling from sample analysis to data interpretations.
4. To determine the concentration of BPA in different types/source of thermal paper.

3. Experimental Apparatus

- Label
- Scissors/puncher
- 20mL plastic/test tube
- 25mL volumetric flask (standard solution), 5 units
- Micropipette
- HPLC sample vials
- Water bath sonicator

4. Reagents and Chemicals

- Extraction solvent: HPLC grade methanol, ethanol, ultrapure water
- Mobile phase: acetonitrile (HPLC grade) and ultrapure water
- BPA stock solutions (100ppm)

5. Sample

- 8 different types of thermal paper

6. Procedure

All glassware used for electrochemistry should be as clean as possible. The solvents and reagents used to make solutions should be as pure as possible. It is a good idea to use deionized, ultrafiltered (DIUF) water or “conductivity water” or “HPLC grade water” for the final rinsing of glassware and for all solution preparation.

A. CALIBRATION STANDARD SOLUTION PREPARATION

Each group of students should prepare five standard solutions with various concentrations ranging from 0 to 25ppm from 100ppm of BPA stock solution. The solutions can be prepared by pipetting various volumes of the stock solution into a series of five 25 mL volumetric flasks. Fill up the concentrations data in the table below:

Standard concentration (ppm)	Flask volume (mL)	Pipette volume (mL)
	25	
	25	
	25	
	25	
	25	

B. OPTIMIZATION OF METHOD

The optimization process was carried out using one thermal paper by varying extraction solvent used (methanol, ethanol and ultrapure water) and extraction duration (5, 10 and 15 minutes) in order to determine the most effective and efficient method and procedure before conducting the experiment for the rest of the samples. The suitable solvent and running time for extraction were determined according to greatest area peak obtained for BPA.

C. SAMPLE PREPARATION

1) Cut or punched each thermal paper (samples) at the printed area using paper puncher/scissor.
2) Weight 20 mg of each sample using electronic balance and place into 20mL plastic/test tube.
3) Add the identified solvent during the optimization process into the plastic/test tube before.
4) Put all samples in a water bath sonicator at selected extraction time obtained from optimization process.
5) The samples were then filtered into the HPLC vials using 0.20 μm syringe filter.

* Each sample should be prepared in triplicated

References

1. Yalcin, M. S., Geçgel, C., & Battal, D. (2016). Determination of bisphenol A in thermal paper receipts. Journal of the Turkish Chemical Society, Section A: Chemistry, 3. doi:10.18596/jotcsa.21345
2. Shimadzu LC-10/20 System Clarity Control Module. (2020). Data Apex, Prague, The Czech Republic.
3. Meyer, V. R. (2010). Practical high-performance liquid chromatography (Fifth Edition). A John Wiley and Sons, Ltd., Publication

Experiment 3

THE DETERMINATION OF HEAVY METALS BY ACID DIGESTION SYSTEM AND FLAME ATOMIC ABSORPTION SPECTROSCOPY (FAAS)

1. Synopsis

This experiment is designed to acquaint in student with the techniques of FAAS for the analysis of metals. The use of an FAAS is illustrated for the determination of metals in different types of tea leaves and/or tea leaves products.

The goals of this experiments are to be familiar with 1) stock and calibration standard preparation, 2) sample preparation, 3) FAAS application, 4) sample analysis & data interpretations, and 5) report writing.

2. Objectives [List down your objective(s)]

E.g: To determine the concentration of heavy metals, Magnesium (Mg), Copper (Cu), Zinc (Zn) and Lead (Pb) in different types of tea leaves such as Black tea, Oolong tea, Green tea and Puer tea by acid digestion method using microwave digestion technique and flame atomic absorption spectroscopy (FAAS).

3. Experimental Apparatus [List down all of the apparatus]. E.g:

- E.g: 250mL beaker
-

4. Reagents and Chemicals [List down all of the reagents and chemicals]

- E.g: Concentrated nitric acid
-

5. Sample [List down all of the samples selected]

- E.g: Tea leaves

6. Procedure [List down all of the procedures/methodology]

*All glassware used for electrochemistry should be as clean as possible. The solvents and reagents used to make solutions should be as pure as possible. It is a good idea to use deionized, ultrafiltered (DIUF) water or “conductivity water” or “HPLC grade water” for the final rinsing of glassware and for all solution preparation.

References [List down all of references]

Experiment 4

ANALYSIS OF TRACE METALS IN MILK BY FLAME ATOMIC ABSORPTION SPECTROPHOTOMETRY (FAAS)

1. Synopsis

Milk is a good source of calcium (Ca), magnesium (Mg), phosphorus (P), potassium (K), selenium (Se), and zinc (Zn). This experiment is designed to acquaint in student with the techniques of FAAS for the analysis of metals. The use of an FAAS is illustrated for the determination of the Ca, Mg, Se and Zn in all types of milk and/or milk products.

2. Objectives

At the end of this experiment, students should be able to apply FAAS for the analysis of Ca, Mg, Se and Zn in milk and/or milk products.

3. Experimental Apparatus

- 250mL beaker
- Digestion microwave
- Glass cover/ watch glass
- Label
- Sterile Plastic/sample bottles
- 100mL volumetric flask (stock solution 1000ppm & 100ppm)
- 25mL VF (standard solution), 5 units
- Micropipette

4. Reagents and Chemicals

- Ca, Mg, Se and Zn standard solution (single element)
- Acids for milk and/or milk products sample digestion

5. Sample

- Milk

6. Procedure

All glassware used for electrochemistry should be as clean as possible. The solvents and reagents used to make solutions should be as pure as possible. It is a good idea to use deionized, ultrafiltered (DIUF) water or “conductivity water” or “HPLC grade water” for the final rinsing of glassware and for all solution preparation.

A. SAMPLING

Three types of milk and/or milk products that are sold in local supermarket will be selected for Ca, Mg, Se and Zn analyses using FAAS. The purchased milk products are then stored in a fridge/chiller (4°C) for sample perseverance before the experiment being executed.

B. MICROWAVE DIGESTION

The milk and/or milk products samples that have been weighed (0.2 g) are put into a clean Teflon microwave vials using a plastic spatula. A mixture of acids (with total volume 10 mL) (follow the default guideline based on sample type given by the microwave digester) is added into the vials. The vials are then put into the microwave rack, then only is placed into the microwave digester.

C. PREPARATION OF DIGESTED SAMPLES

The products of the digestion are filtered using filter paper Whatman no.1 and put into 50 mL volumetric flask. Then, the solutions are topped up until 50 mL graduation mark with deionized water. The solution is then put into a plastic container to prevent any metals from sticking to the internal glass wall of the volumetric flask.

D. PREPARATION OF STANDARD STOCK SOLUTION

Standard for each element (Ca, Mg, Se and Zn) with concentration of 1000 ppm can be obtained from lab (kindly approach Encik Mohd Hazni). The standard can then be diluted with deionized water to 3.0, 3.5, 4.0, 4.5, 5.0 ppm (or any concentration range that suit your tested samples), respectively with 50 mL volumetric flask.

Experiment 5

AN INTRODUCTION TO CYCLIC VOLTAMMETRY

1. Synopsis

For several decades, cyclic voltammetry has been a very popular and often used electroanalytical technique. A cyclic voltammogram (or CV) is obtained by applying a linear potential sweep (that is, a potential that increases or decreases linearly with time) to the working electrode. As the potential is swept back and forth past the formal potential, E^o, of an analyte, a current flow through the electrode that either oxidizes or reduces the analyte. The magnitude of this current is proportional to the concentration of the analyte in solution, which allows cyclic voltammetry to be used in an analytical determination of concentration.

The goal of this experiment is to become familiar with using a modern electrochemical potentiostat, to determine the concentration of $Ru(NH_3)_6Cl_3$, 99%, in an unknown solution, and to measure the diffusion coefficient for the hexaaminеruthenium(III) cation, $Ru(NH_3)_6Cl_3$. This procedure illustrates how the current observed in a cyclic voltammetry experiment depends upon experimental parameters such as concentration and sweep rate.

2. Objectives

e) To study the effect of concentration and sweep rate on the peak height and peak potential.
f) To calculate the diffusion coefficient for the hexaaminеruthenium(III) cation based on the slope of the calibration plot generated.
g) To determine the concentration of unknown solution using the equation generated from the calibration plot.

3. Experimental Apparatus

- Metrohm Autolab PGSTAT
- Metrohm NOVA software
- Metrohm Three Electrode Cell
- Four 25 mL volumetric flasks, one 250 mL
- Pipettes

4. Reagents and Chemicals

Description	per expt
Supporting Electrolyte 1.0 M potassium chloride (KCl)	250 mL
Analyte Stock Solution 6.0 mM potassium $Ru(NH_3)_6Cl_3$ prepared using 1.0 M KCl as the solvent	25 mL

5. Procedure

All glassware used for electrochemistry should be as clean as possible. The solvents and reagents used to make solutions should be as pure as possible. It is a good idea to use deionized, ultrafiltered (DIUF) water or "conductivity water" or "HPLC grade water" for the final rinsing of glassware and for all solution preparation.

A. SOLUTION PREPARATION

1) These stock solutions should be provided to a group of 2-3 students:

1.0 M potassium chloride (250 mL)
[KCl formula mass is 74.56 g mol^{-1}; CAS number 7447-40-7]

Saturated solutions of potassium chloride available directly from chemical manufacturers may be diluted and used as electrolyte solutions for cyclic voltammetry. Alternately, the potassium chloride solution can be prepared by dissolving _____ grams of KCl in enough ultrapure water to obtain a 250 mL final volume.

6.0 mM $Ru(NH_3)_6Cl_3$ stock solution (25 mL)
[$Ru(NH_3)_6Cl_3$ formula mass is 309.6121 g mol^{-1}; CAS number 14282-91-8]

This stock solution can be prepared by dissolving ____ milligrams of $Ru(NH_3)_6Cl_3$ in enough 1.0 M potassium chloride solution to make 25 mL of solution. The resulting solution has a $Ru(NH_3)_6Cl_3$ concentration near 6.0 mM.

2) Each group of students should prepare four standard solutions with various concentrations of $Ru(NH_3)_6Cl_3$ solution ranging from 0.5 to 6.0 mM. One of the standard solutions should be the stock solution itself (6.0 mM). The other three solutions can be prepared by pipetting various volumes of the stock solution into a series of three 25 mL volumetric flasks. When filling each flask "to the line," be sure to use the 1.0 M potassium chloride solution rather than deionised water. The table below is meant to serve as a guide in making these three solutions. The concentrations listed in the table assume that the $Ru(NH_3)_6Cl_3$ stock solution has a concentration of 6.0 mM. Students should verify this assumption and compute the concentrations again if needed.

Pipette volume (mL)	Flask volume (mL)	Standard concentration (mM)
2.5	25	0.6
5	25	1.2
12.5	25	3.0

B. SETTING UP THE SOFTWARE

6) Create a new file in ‘Documents’

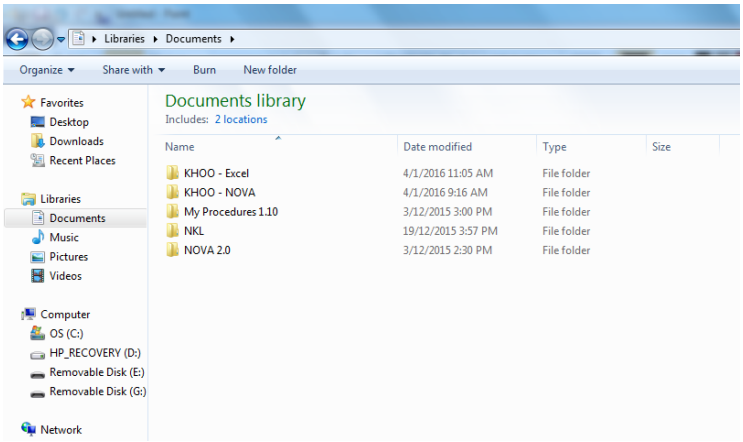


Figure 1.1

Example: KHOO – NOVA

7) Click the ‘NOVA’ icon on the desktop.

8) Open the created file (KHOO – NOVA)

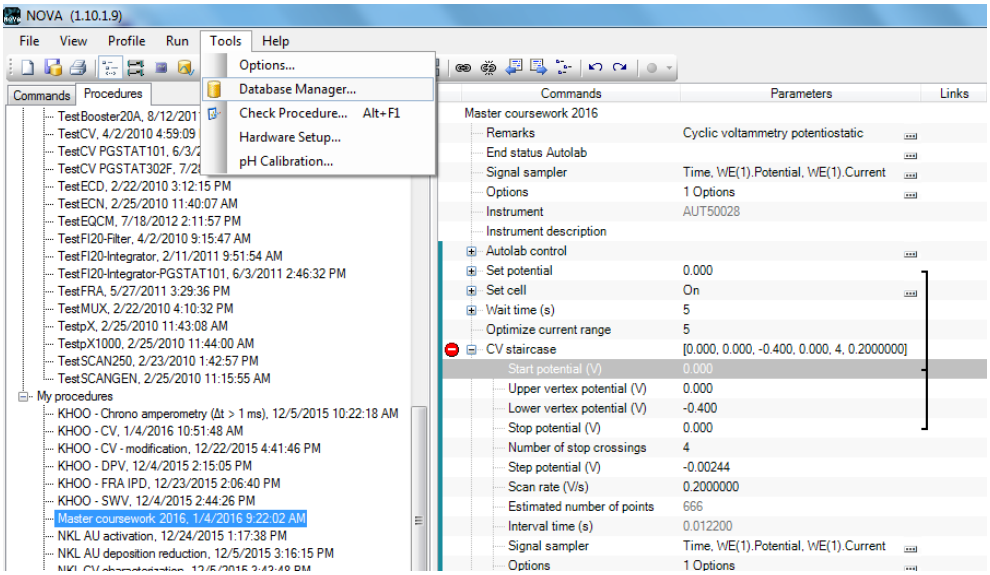


Figure 1.2

Click ‘Tools’ and then ‘Database Manager’

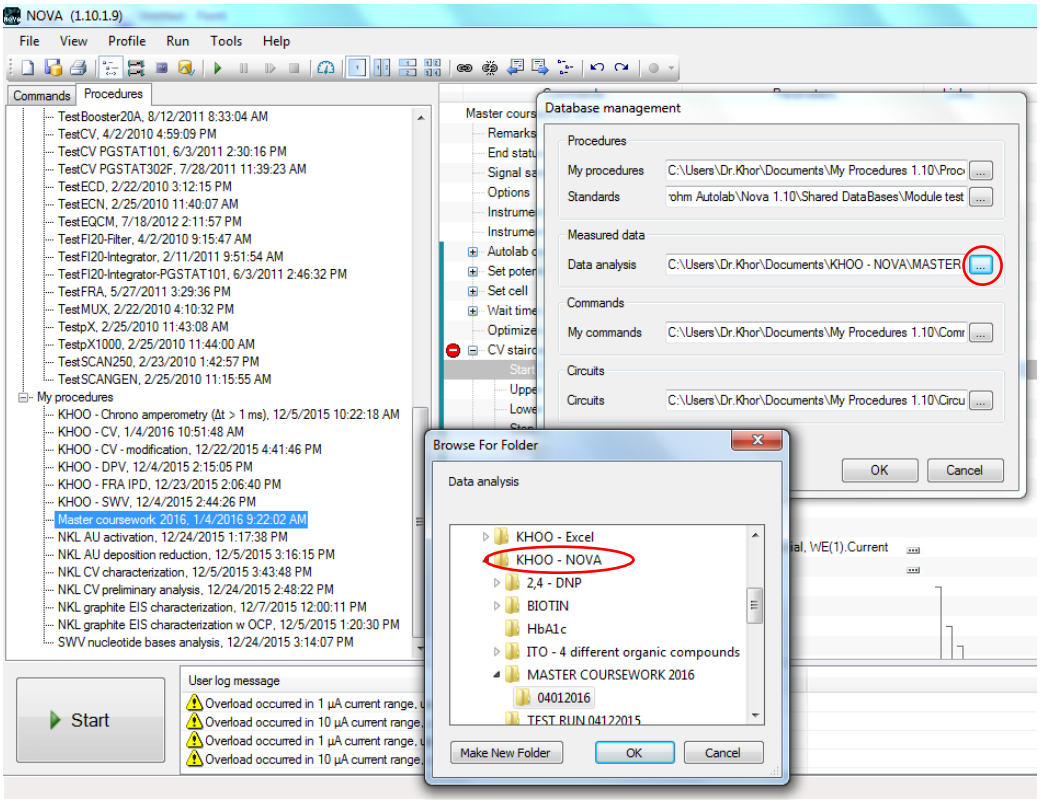


Figure 1.3

Click ‘…’ icon behind the ‘Data Analysis’ and then select the created file.

9) Experiment setting

Click  icon in the menu bar, and then select the ‘Cyclic Voltammetry Potentiostatic’ from ‘Procedures’.

Change the setting according to **Figure 1.4**.

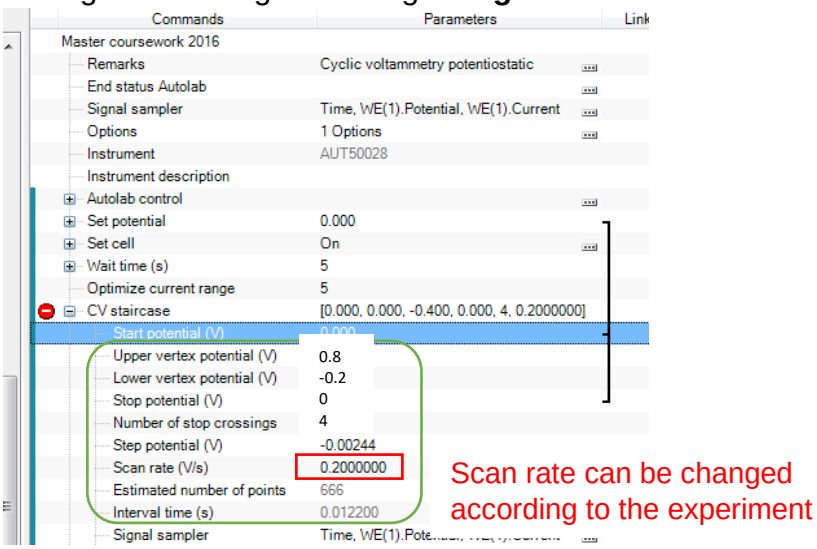


Figure 1.4

10) Make sure all the electrodes are connected properly. Then, click ‘START’.
Red banana plug is connected to the working electrode.
Black banana plug is connected to the auxiliary electrode.
Blue banana plug is connected to the reference electrode.

11) To rename the cyclic voltammograms and to do analysis.

Click  icon in the menu bar.

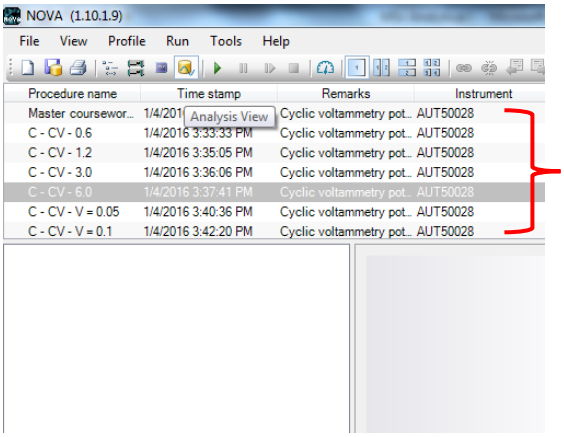


Figure 1.5

Rename: Right click on the measurement that you want to rename, select ‘Properties’, then change the name in the space.

Analysis (comparison, peak height, …): Double click on the measurements that you want to analyse, after that it will appear in the box below (left).

a. For comparison, press ‘ctrl’ and click on the ‘i vs E’.

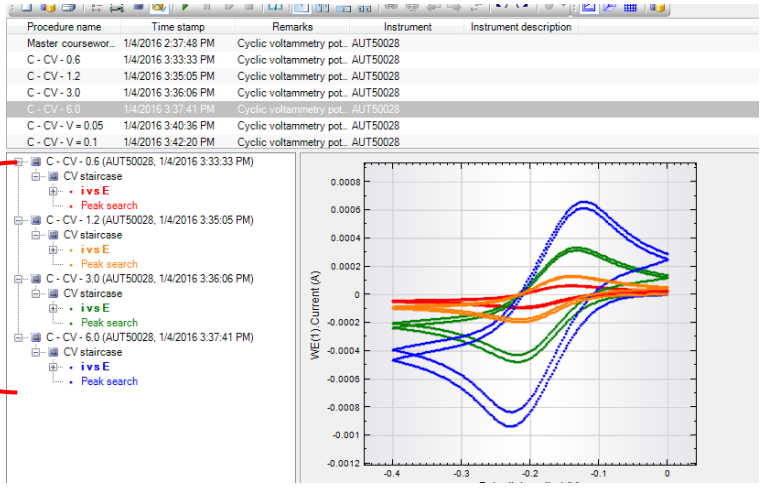


Figure 1.6

b. For peak height, right click on ‘i vs E’ and follow the steps in **Figure 1.7**.

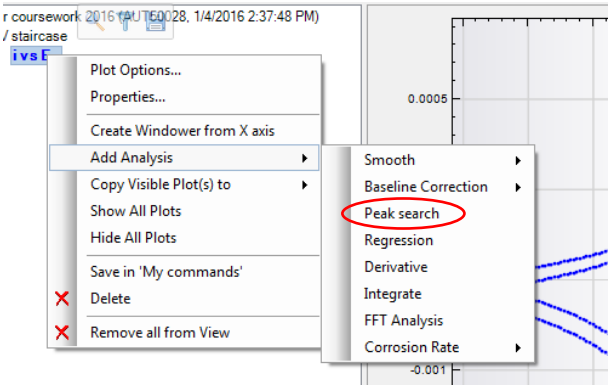


Figure 1.7

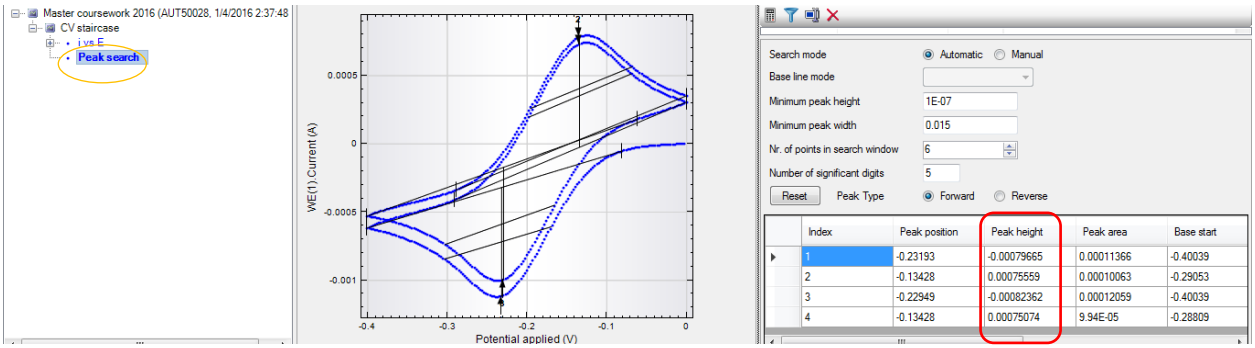


Figure 1.8

Peak height

C. THE EFFECT OF CONCENTRATION

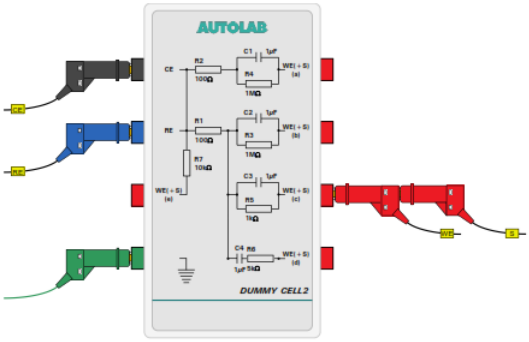
In this part of the experiment, several cyclic voltammograms are obtained with solutions of varying concentration. Four standard solutions with concentrations ranging from 0.6 to 6.0 mM should be used. The standards should be prepared using 1.0 M potassium chloride solution as the solvent.

1) Obtain a clean 20 mL glass vial.

2) Fill the glass vial with 5.0 mL of the standard solution which has the lowest $Ru(NH_3)_6Cl_3$ concentration.

3) Place the working electrode, auxiliary electrode, and the reference electrode inside the vial. Make sure that all three electrodes are immersed in the solution.

4) Before making electrical connections between the cell and the potentiostat, it is a good idea to make sure the Autolab PGSTAT in DUMMY mode. This can be done by connecting the cell cables to Dummy cell as figure below. Next, choose the ‘Cyclic Voltammetry Potentiostatic’ from ‘Procedures’, then click ‘START’.



Dummy cell connections

Figure 1.9

5) Use the special cable to connect the potentiostat to the working, auxiliary and reference electrodes.

6) After the cell has been properly configured, change the setting in the NOVA software according to **Figure 1.4**. Then, click 'START'.

7) A fairly prominent cathodic wave should appear during the sweep from 0 mV to -400 mV. On the return sweep, an anodic wave of roughly equal size should appear.

8) After acquiring a satisfactory voltammogram, name the measurements and do analysis (comparison, peak height) according to **Step 8** (To rename the cyclic voltammograms and to do analysis).

9) To obtain 'i vs t' plot, follow then steps in **Figure 1.10**.

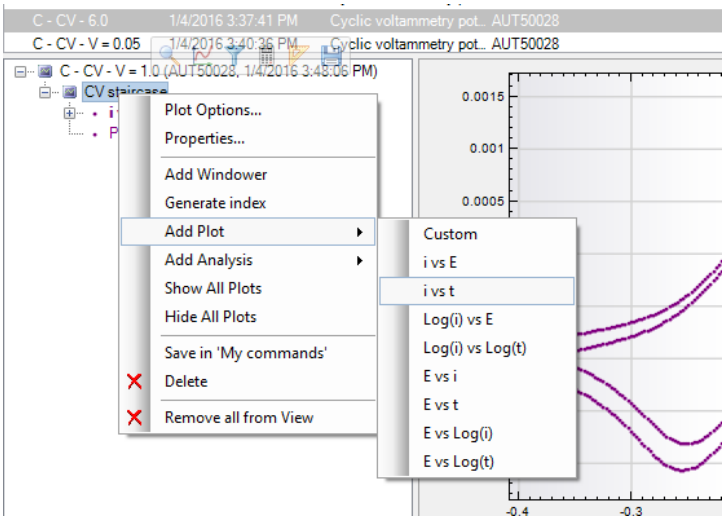


Figure 1.10

10) Acquire similar voltammograms for the remaining standard solutions in order of increasing concentration. Use the same sweep rate (200 mV s^{-1}) for all solutions. Clean the electrochemical cell using 1.0 M potassium chloride solution between standard solutions.

11) After the last standard solution (6.0 mM) has been studied, leave it in the cell and use it for the next part of the experiment.

D. THE EFFECT OF SWEEP RATE

Using the standard $Ru(NH_3)_6Cl_3$ solution with the highest concentration (6 mM), a series of cyclic voltammograms should be acquired at various sweep rates. The peak current observed in these voltammograms should exhibit a noticeable dependence on the sweep rate.

1) Using sweep parameters like those in **Figure 1.4**, acquire voltammograms at the following sweep rates: 50, 100, 200, 500, and 1000 mV s^{-1}. In addition, the height of the cathodic peak from each voltammogram can be obtained by following the steps in **Figure 1.7**.

E. THE UNKNOWN SOLUTION

The instructor may provide a solution which has an unknown concentration. This solution should be examined using the same sweep rate as that used to examine the series of standard solutions in Part C of this procedure (200 mV s^{-1}). Because the unknown solution has a concentration on the same order as your standard solutions, it may be examined directly as supplied by the instructor.

1) Using sweep parameters similar to those in **Figure 1.4**, acquire a voltammogram of the unknown solution using a 200 mV s^{-1} sweep rate. In addition, the height of the cathodic peak from each voltammogram can be obtained by following the steps in **Figure 1.7**.

6. Data Analysis

Concentration Study

a) Using the cathodic peak currents for the series of standard solutions, prepare a plot of peak current versus concentration. (Make sure that each of the voltammograms was acquired using the same sweep rate.)

b) Perform a linear least squares analysis on the data to find the equation of the best straight line which fits the data.

c) Use the slope of the line to calculate the diffusion coefficient for the $Ru(NH_3)_6Cl_3$ cation.

Unknown Solution

d) Using the equation from (b), above, compute the concentration of $Ru(NH_3)_6Cl_3$ cation in the unknown solution. Report your result in moles per liter using three significant figures.

Sweep Rate Study

e) Using the cathodic peak currents measured from the series of voltammograms acquired at different sweep rates, prepare a plot of peak current versus the square root of the sweep rate. (Make sure that each of the voltammograms was acquired using the same standard solution.)

f) Perform a linear least squares analysis on the data to find the equation of the best straight line which fits the data.

g) Use the slope of the line to calculate the diffusion coefficient for the $Ru(NH_3)_6Cl_3$ cation.

References

4. Scholz, Fritz (2010) Electroanalytical Methods [electronic resource]: Guide to Experiments and Applications. Berlin, Heidelberg: Springer Berlin Heidelberg: Imprint: Springer.
5. Wang, Joseph (c2006) Analytical electrochemistry. Hoboken, N.J.: Wiley-VCH.
6. Lewis, Jaylen (2012) Encyclopaedia of electrochemistry: principles and applications. London: Auris Reference