Introduction to Analytical Glassware and Procedures

Before lab:

Read lab, watch video
Perform calculation section D, question 1

Introduction:

This lab exercise is designed to familiarize you with analytical glassware and equipment, and introduce the concepts of accuracy, precision, and error. Every measurement you make has error associated with it. For analytical chemistry, where the goal is to make measurements that are both accurate and precise, it is essential to understand the magnitude of error associated with different measurements.

During this lab you will have the opportunity to use the glassware you will be using for the rest of the semester. It is essential that each student take the opportunity to play with all the glassware because you will be graded on the accuracy and precision of your experimental results (only achieved through careful and proper use of glassware) throughout the semester. A short description of the analytical equipment you will be using today is provided below. Be sure to familiarize yourself with the terminology before coming to lab.

Manual pipettes: used to transfer liquid with more precision than a graduated cylinder, they are thought to be relatively precise and accurate especially if calibrated before use.

Automatic pipettes: used to transfer liquid with more precision than a manual pipette can offer and if calibrated well, is one of the most precise and accurate methods available for dispensing liquids

Volumetric flasks for making stock solutions: widely used to prepare stock solutions, typically requires pipetting some volume of liquid (using a manual or automatic pipette) and filling the remaining portion of the volumetric flask with DI water or some other reagent until the bottom of the meniscus is level with the line located on the neck of the flask.

Procedure and Data Collection:

In this lab, you will work in pairs and visit different stations set up with all the appropriate glassware and equipment needed to collect that portion of data. If the station requires using a balance, all balances are located together by the glassware cabinet. All groups need to visit each station outlined below.

Station A: Automatic and manual pipet delivery volumes

Purpose: measure the accuracy and precision of automatic and volumetric pipet delivery volumes.

Procedure:

1. Record empty beaker tare mass
2. Using either pipet, pipet 10 ml into the beaker
3. Weigh beaker with 10 ml H2O on an analytical balance and record mass
4. Repeat 10 times.

Results:

Beaker tare mass:

Replicate measurements	Automatic pipette	Manual pipette
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		

Station B: Measuring 100 mL

Purpose: determining which glassware most accurately and precisely measures volume.

Procedure:

1. Record empty beaker tare mass
2. Measure 50 mL as accurately as you can into a beaker
3. Weigh beaker with 50 ml H_2O on an analytical balance and record mass
4. Repeat 5 times for each piece of glassware

Results:
Beaker tare mass:

Replicate measurements	Automatic pipette	Volumetric flask	beaker	Graduated cylinder
1				
2				
3				
4				
5				

Station C: Precision of an analytical balance

Purpose: Compare the accuracy and precision of an analytical and top loader balance.

Procedure:
1. Record the mass of a single nickel on top loader balance. Repeat 10 times.
2. Record the mass of the same nickel on analytical balance. Repeat 10 times.

Results:

Replicate	Top loader	Analytical
1		
2		
3		
4		
5		
6		
7		
8		
9		
10		

Station D: Making a stock solution of 0.2 M $NiSO_4 \cdot 6H_2O$

Purpose: Make a stock solution to use for serial dilutions next lab.

Procedure:
1. Calculate the mass of $NiSO_4 \cdot 6H_2O$ required to make 50 ml of 0.2 M solution
2. Tare volumetric flask
3. Weigh $NiSO_4 \cdot 6H_2O$
4. Quantitatively (without loss of ANY solid) transfer to the volumetric flask and dilute to volume.

5. Weigh total solution in volumetric flask
6. Label the vial and store it for next week

Results:

Measurement	value
Tare mass	
Mass $NiSO_4 \bullet 6H_2O$	
Total mass	

Station E: Enter your data from station B and C into the computer.
Procedure:
Enter your data for Stations B and Station C into the lab computer. BE SURE TO SUBTRACT THE TARE WEIGHT FROM THE VALUES. You can email yourself a copy of your own data to work on later.

Calculations and Discussions:

Station A:
Calculate the average and standard deviation of your measurements.

Discussion Questions:
Q1: Which method is more accurate and precise? How do you know?

Q2: Compare your automatic pipet measurements with the published allowable tolerances. http://www.johnmorris.com.au/files/files/PDFs/Gilson/Maximum%20errors%20tolerated.pdf.

Station B:
Calculate the average and standard deviation of your measurements.

Discussion Questions:
Q1: List measurement methods in order from most to least precise and accurate. Do these results conform to your expectations?

Q2: Give three possible reasons you might use a less precise method for measuring volume.

Station C:
Calculate the average and standard deviation of your measurements. For the analytical balance measurements calculate the relative percent errors of your measurements.

Discussion Questions:
Q1: What is the error associated with an analytical balance? Does this surprise you?

Station D:
Discussion Questions:

Q1: Describe in detail (including what glassware and balances you used) how you performed this procedure.

Q2: Calculate the exact molarity of the solution you made.

Q3: What, given the tools available to you, is the most accurate method of preparing solutions?

Q4: List three potential sources of error in making solutions

Whole Class Data Exercises: Using data from the entire class

1. Calculate the average and standard deviation comparing measurements of 100 ml using the automatic pipettes, volumetric flasks, beaker, and graduate cylinder (Station B). How the accuracy and precision of your measurements compare with the measurements of the entire class? Perform a Student's t-test to determine if there is a statistical difference between your mean and the whole class mean with a 90% confidence interval. Do not use your data in the "whole class" data you are comparing against.

2. Calculate the average and standard deviation of the mass of the nickel for the entire class. How does this compare to values reported by the US Mint.
   http://www.usmint.gov/about_the_mint/?action=coin_specifications
   Perform a Student's t-test to determine if your value is significantly different from the published value.

3. In 2-3 sentences, tell me how your group worked for this lab. What worked well? Did everyone contribute? Quantify relative contributions in the table below.

Group member name	Percent lab contribution

Lab Write-up Requirements:

Lab reports need to be typed and turned in at the beginning of lab the week following the experiment (**late work is not accepted!**). For full credit, a lab report will be neat, organized, legible, and contain the following sections:

1. **Purpose-** One sentence stating the purpose of the experiment
2. **Calculations-** Show one hand-written example calculation for each type of calculation (eg. One average, one standard deviation). Use Excel or similar to calculate the remainder.
3. **Questions-** Address the questions for each station and the whole class data.
4. **Appendix 1: Raw data-** Put the experiment sheet at the end of the lab report to show your raw data.