Practice. Balances and calibration of volumetric tools

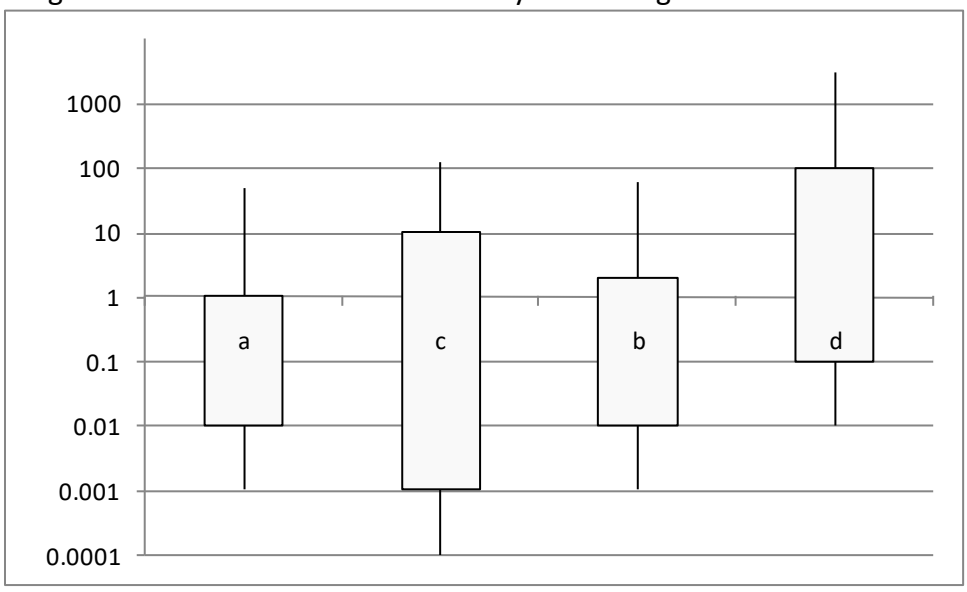
Balances are a very basic and very valuable tool in any chemistry lab and any chemist must understand their use, their proper treatment, their strengths and their vulnerabilities.

Dynamic ranges

The precision of a modern balance can be quite astounding, but their *dynamic range* (the ratio between the smallest and largest weight they can handle) is limited. This is why they come in different ranges. Some measure in tons, others in kg, yet others in grams, in mg or even in micrograms. In the lab we generally use two kinds of instruments:

- gram range (ca. 1000g down to ca .01 gram)
- milligram range (ca. 50g down to ca 0.1 mg)

The graph shows the dynamic ranges of the balances in Dabney 608 on a logarithmic scale in grams. It is up to you to *choose* the appropriate device for your measurement. Ideally your weight should be in the middle of the dynamic range



If a mass of 1 gram is weighed on a balance that goes down to .01 grams the *relative error* = .01/1 = 1%
If the same mass is weighed on a balance going down to .1 mg the relative error is .0001/1 = 0.01%
At the top of the dynamic range the precision gets even better, but then the accuracy is often no longer guaranteed.

Vulnerabilities

1. Air flow can affect measurements in the mg range
2. Buoyancy can affect measurements in the sub-mg range
3. Vibrations can affect measurements in the mg range
4. If the balance is not properly leveled this can affect the weight.
5. Suddenly dropping a heavy object of severe overloads can destroy the balance
6. uneven spread of the weight may lead to erroneous values
7. Leaving spilled chemicals on the balance corrodes it (at all weight ranges). ***CLEAN THEM UP!!!!***
8. To avoid corrosion, always use paper / aluminum / ceramic /glass containers or weighing boats.
9. Hygroscopic materials gain weight if left open. Either weigh quickly or weigh them in a flask you can close off

Introduction

Balances need calibration with standard weights, but once calibrated, they are used to calibrate other equipment. The purpose of this lab is (1) to calibrate two volumetric tools (a pipette, and an automatic pipette) and (2) to determine the density of a liquid using a burette. Ethanol or other liquids will be provided by your TA.

You will then use the Reweighted Least Squares technique (1D) to reject any outliers and calculate the average and RMSE of the volume delivered by the automatic pipette (micropipette) and the burette, respectively.

You will also use the Reweighted Least Squares technique (2D) to reject any outliers and calculate the density of the liquid from the burette measurements.

The dimension of measurement of the pipettes and graduated cylinder is *volume* rather than mass. This is why we need to know the *density* to be able to convert between the two:

mass [g] = volume [ml] * density [g/ml]

The density of a liquid depends a little on temperature. Therefore, before the measurement use a thermometer to *measure the temperature* of the liquid you are using and look up the density at that temperature. Compare the density determined from the burette measurements to the literature density of the liquid under the same temperature condition.

Experiment.

Caution: If you spill anything: clean up immediately, just use Kim wipes. Please do not ruin balances by leaving them dirty or wet! Don't push hard on a balance.

The basic procedure is essentially the same for the three volumetric instruments. Make sure you first properly rinse the volumetric tool with DI water then condition it with the liquid. There is DI a tap in the corner of Dabney 608 by the white board.

Write down all information about your volumetric tool: what volume can it measure? Do you know the tolerance (uncertainty)?

Select a suitable balance. Which one is suitable depends on the quantities you are about to work with, so you need to compare the weight you expect to get with the dynamic range of the balance. Take as sensitive a balance as you can afford under the circumstances. Use an appropriately sized beaker as the receptacle for weighing.

The measurement should be done using ten aliquots should be dispensed from your device consecutively.

There are few different strategies to weigh a sample:

1. You can either let the liquid accumulate in your weighing pan and tare each time you wish to dispense more
2. You can discard the liquid each time and then tare.
3. You can let the weight accumulate and not tare at all (keeping careful records of the values at each accumulation)

The last measurement may result in having to deal with greater quantities. (Is your balance capable of dealing with that?)
Make sure you write down the weight of the dispensed aliquot (or the total weight in strategy 3) in your lab notebook.

For the a**utomatic *pipette*** (micropipette)
Set the variable volume pipette to each volumetric value and write down which value you are measuring. For the calibration step, use strategy 2. above to record at least 5 different readings of the mass of the liquid delivered by your selected pipet at each volume. For the measurement of the density measure each of ten volumes ranging from 0.1 to 2.0 mL. Measure each volume and mass in triplicate.

For the ***burette***:
The burette will only be used for the density measurement using strategy 1. above. Fill your burette and record the initial volume (if you do not start at exactly zero, you will have to subtract this initial reading from all the subsequent readings). Using weighing strategy 3. above, tare a clean dry receiver flask and deliver approximately 1 ml aliquots (record the exact volume-bottom of the meniscus) and record the mass on the analytical balance after each delivery. Do not tare between measurements

Data work-up

Put your data in a spreadsheet. Make sure to convert the liquid masses to volumes with the right density for the pipettes. If you did accumulation compute the difference between a weight and the previous to determine the weight of each aliquot.

Determine the Relative and Systematic Error of the Micropipette

Use the Reweighted Least Squares technique to reject any outliers and calculate the average volume of the pipette and its uncertainty. This is like a calibration step, except that you may use a single volume, e.g. 1 mL for the large micropipette or 200 uL for the smaller variant and weigh a given volume of DI water at least three times. Based on the 95% confidence limit, what is the relative error? Relative error refers to the ratio of the 95% confidence limit to the nominal value used and may be converted to percent.

Also determine the ***systematic*** error of the pipette (the nominal value written on it minus your final average). Is this bias significant? Is there a tolerance given on the pipette? Is the bias within that tolerance?

Pipettes are often used in the first step of a dilution. Suppose you would use your pipette to dilute an aliquot of a 0.10000 molar solution to a volume of 1 liter in a volumetric flask. Calculate the final concentration of that solution *and* its precision (2/15 please) assuming that the molarity 0.10000 and final volume of 1000 ml are exact (error free). What is the relative error in the concentration?

Determine the Density of a Liquid Using the Micropipette and Burette

Graph ten measured weights against ten different volume values ranging from 0.1 to 2.0 mL that you read off from 1.) the micropipette and 2.) the burette. Do a regression (RLS if necessary). The slope value should equal the density of the liquid at your temperature. Use the trumpets Excel spreadsheet to calculate the slope, RMSE and 95% confidence limit for a line of regression. A. Is the difference in the measured and tabulated values of the density within precision? B. What is the RMSE of the slope of the line of regression? C. What is the t-value? D. Using the graphical method draw a horizontal line at a mass of 1 gram and determine the 95% confidence limit.

General questions

The accumulation method and the taring method each have advantages and disadvantages. The taring method limits the total weight, and that can be an advantage. It also has a drawback. Which one?

Laboratory Report

This laboratory is an exercise in measurement and calibration. Find articles in the Journal of Chemical Education or journals in Statistics, Physics or other quantitative sciences that describe

methods for calibration and error determination. Write the paper as a general introduction to these methods using the categories in the rubric, Abstract, Introduction, Experimental, Results and Discussion. Discuss relative errors in measurement of density based on your data and any articles. Also, consider the errors in measurements of pH, heats of combustion, concentration (based on absorbance for example) or any other commonly measured quantity. Is there a consensus about how large a relative error is commonly observed or is acceptable for a scientific measurement?