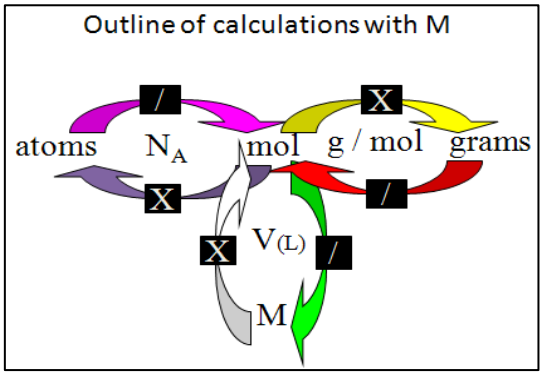
Name _____ **TA** _____ **Time** _____

An Introduction to Titrations

A titration is an experiment used to determine the concentration of an unknown solution. Titrations can be used with several types of reactions such as oxidation/reduction reactions or other more complicated reactions in order to determine how much of something is in the solution to be analyzed. This introduction will use three simple acid base reactions to illustrate the principles of a titration experiment.

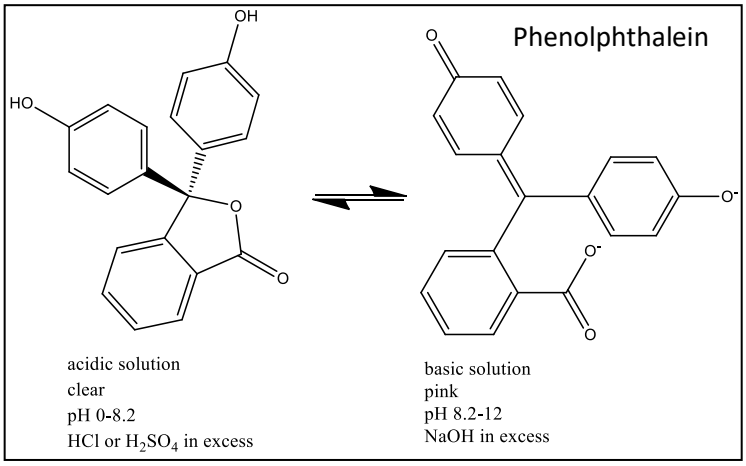
Theory



All solutions used in this experiment will use the term molarity, M, which is defined as the moles of solute (the lesser quantity) divided by the volume of the solution (solute + solvent) in liters, L. Therefore anywhere you see "M" you can replace "M" with mol/L. Molarity fits in with the other stoichiometric calculations as shown.

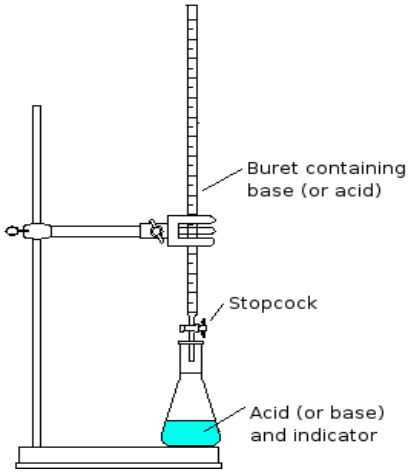
In this experiment you will attempt to determine the molarity of two unknown solutions one containing HCl and the other H_2SO_4. To do this, you will determine the concentration in molarity of a NaOH solution as the standard. You will use this known concentration to determine the concentration of both of your unknowns. An acid added to a base will produce a salt and water.

You will take a sample of your acid and add base to it until all of the acid has been reacted. In order to know when the acid has all been reacted you will add an indicator.



Indicators are usually organic molecules which are colored under certain conditions or may change colors as the conditions change. They can detect changes in oxidation states, presence of particular substances or like in our case the presence of acidic / basic conditions.

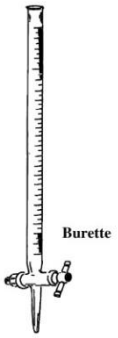
Phenolphthalein is the indicator you will use in this lab, a Lewis structure of phenolphthalein is shown depicting its structure in both acidic and basic solutions.



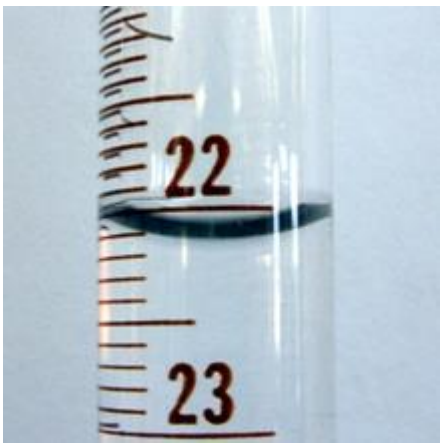
Because the Phenolphthalein is clear in acidic solution and pink in basic solution when you have added enough NaOH to one of your acid samples to turn the solution pink you have gone past the **equivalence point** where moles of acid equals moles of base to the **endpoint** which is the physical change (color change) which best approximates the equivalence point. Therefore you are trying to stop your titration at a point where it is the lightest pink you can detect. That is the best approximation of the

equivalence point. In this experiment you will be asked to dilute an acid. You always add the acid to the water because there is heat generated and the water can absorb the heat. The reverse can result in the acid boiling and spattering.

Equipment you will use



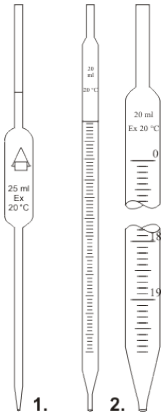
The NaOH solution will be dispensed from a burette. It has markings in the opposite direction of a graduated cylinder. You need to completely clean the burette with soap and water, then wash with distilled water, and then rinse twice with a small amount of the solution you will use, NaOH. Then fill the burette close to but not over the "0" mark and read it accurately to the nearest 0.02 mL, you can do this by estimation. Use the thickness to the line to be 0.02 mL and estimate how many lines you could draw to reach the bottom of the meniscus. (The example on the right should be read as 22.12 mL). Pipettes are used to deliver a precise volume of liquid they have one mark and the liquid is drawn until the bottom of the meniscus is on the mark using the vacuum bulb shown in the drawing. DO NOT ALLOW ANY LIQUID INTO THE BULBS IT WILL RUIN THEM!



Calculations

For any dilution use the equation $\mathbf{M_1V_1 = M_2V_2}$

If your reaction is a 1:1 ratio of stoichiometric coefficients in the balanced equation, you can use the dilution equation to calculate the molarity of the unknown acid because moles of acid = moles of base. This is true for the HCl and NaOH reaction and the HNO_3 and NaOH reaction.



If your reaction is NOT a 1:1 ratio of stoichiometric coefficients in the balanced equation, you CANNOT use the dilution equation directly to calculate the molarity of the unknown acid because moles of acid ≠ moles of base. In this case you have to calculate the moles of base from the molarity given and the volume of NaOH used, then use the stoichiometric coefficients to calculate the moles of acid, and finally use the volume of acid in the sample to calculate the molarity of the acid. Another way to calculate your unknown molarity is to use the ratio for this specific reaction with NaOH and H_2SO_4: $\mathbf{2\ M_{H2SO4}V_{H2SO4} = M_{NaOH}V_{NaOH}}$

The formula for standard deviation is: $s = \sqrt{\frac{\Sigma(\bar{x}-x_i)^2}{n-1}}$ where x_i is the individual calculation, $\bar{x}$ is the average, n is the number of calculations, and Σ is the summation symbol which means "add up what follows."

(*See the instructions on page 6 for using Excel to calculate standard deviations using the formula above.*)

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Experimental

Standardization of the NaOH solution

Clean your burette with soap and water, rinse with tap water, rinse with distilled water and then rinse twice with about 5 mL of the NaOH solution to be standardized. Fill the burette with the NaOH solution to a little below the 0 mark and read it to the nearest 0.02 mL. This is your starting volume. Record it in the table below.

To determine the concentration of the NaOH solution, you will react it with potassium acid phthalate, KHP. It reactants according the equation; KHP + NaOH $\rightarrow$ Na(KP) + H_2O in a neutralization reaction.

Obtain about 20 mL of an approximately 0.10 M KHP solution previously prepared by your TA from one of the burettes located on the demonstration table. Record the burette readings before and after dispensing the solution in a clean 125 mL Erlenmeyer flask. Add 3-5 drops of the phenolphthalein solution and mix. (Your TA will tell you the exact concentration of KHP.)

Titrate your KHP solution with the NaOH solution until a very light pink color results which last at least 30 seconds with stirring. Record the ending volume on the burette to the nearest 0.02 mL. (Your TA will demonstrate the titration.)

Calculate the NaOH molarity, record it and add it to the list on the blackboard. The values will be averaged for the H_2SO_4 and HCl molarity determinations.

H_2SO_4 titration, 1:2 ratio titration

Obtain about 125 mL of unknown H_2SO_4 and pipette exactly 25.00 mL to a clean 250 mL Erlenmeyer flask. Add 3-5 drops of the phenolphthalein solution and mix. Repeat two more times so that three samples of 25 mL H_2SO_4 are ready to titrate.

Again fill the burette to a little below the 0 mark and read it to the nearest 0.02 mL with the standardized NaOH. This is your starting volume.

Titrate one of your H_2SO_4 samples until a very light pink color results which last at least 30 seconds with stirring. Record the ending volume on the burette to the nearest 0.02 mL.

Repeat with your other two samples or until you have three results with reasonable agreement.

HCL titration, titration with a dilution

Obtain 10-20 mL of unknown HCL and pipette exactly 10.00 mL to a graduated cylinder containing 90 mL H_2O. Pour and label the diluted HCl into a clean and dry 125 mL Erlenmeyer flask. Mix the diluted HCl and pipet exactly 25.00 mL to a clean 250 mL Erlenmeyer flask. Add 3-5 drops of the phenolphthalein solution and mix. Repeat two more times so that three samples of 25 mL dilute HCL are ready to titrate.

Again fill the burette to a little below the 0 mark and read it to the nearest 0.02 mL with the standardized NaOH. This is your starting volume.

Titrate one of your dilute HCl samples until a very light pink color results which last at least 30 seconds with stirring. Record the ending volume on the burette to the nearest 0.02 mL.

Repeat with your other two samples or until you have three results with reasonable agreement.

Waste disposal

All solutions that have been titrated can be washed down the sink. You have neutralized your acid samples in the process. Ask the TA to dispose of excess acid and base.

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Part 1

Average Molarity of standardized NaOH _____ (used for all calculations)

NaOH Standardization

Trial #1

Final KHP volume _____mL Final NaOH volume _____mL

Start KHP volume _____mL Start NaOH volume _____mL

Volume KHP _____mL Final NaOH volume _____mL

Use $M_1V_1 = M_2V_2$

Molarity of NaOH _____M Average Class Molarity of NaOH _____M

Part 2

H_2SO_4 titration sample letter _____

Trial	#1	#2	#3	#4
Final volume	_____mL	_____mL	_____mL	_____mL
Start volume	_____mL	_____mL	_____mL	_____mL
Volume NaOH	_____mL	_____mL	_____mL	_____mL
Volume of H_2SO_4	_____mL	_____mL	_____mL	_____mL

$2\ M_{H2SO4}\ V_{H2SO4} = M_{NaOH}\ V_{NaOH}$

Molarity of **H_2SO_4**	_____M	_____M	_____M	_____M

Average molarity _____M

Standard deviation _____

Part 3

HCL titration sample letter _____

Trial	#1	#2	#3	#4
Final volume	_____mL	_____mL	_____mL	_____mL
Start volume	_____mL	_____mL	_____mL	_____mL
Volume NaOH	_____mL	_____mL	_____mL	_____mL
Volume of HCl	_____mL	_____mL	_____mL	_____mL

Use $M_1V_1 = M_2V_2$

Molarity of HCl	_____M	_____M	_____M	_____M

Average molarity _____M

Standard deviation _____

Remember we diluted this sample of HCl so calculate the original molarity of the "concentrated" HCl solution.

_____M

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Questions

1) Why do you want to stop the titration of the solution at the lightest pink color you can detect?

2) How would this experiment change if the HCl was the standard solution and NaOH was the unknown?

3) What is the proper way to dilute acids with water?

4) In this experiment you diluted the HCl given to you and then titrated the samples. What are the possible sources of error in the titration of the HCl samples?

5) Describe how to make 500 mL of 0.435 M HNO_3 from a 6.00 M HNO_3 solution. Do the math and then use wording to describe how to do the dilution.

6) A titration of 50 mL of an unknown concentration of H_2SO_4 required 41.26 mL of 0.1582 M NaOH standard. What is the molarity of the H_2SO_4 solution?

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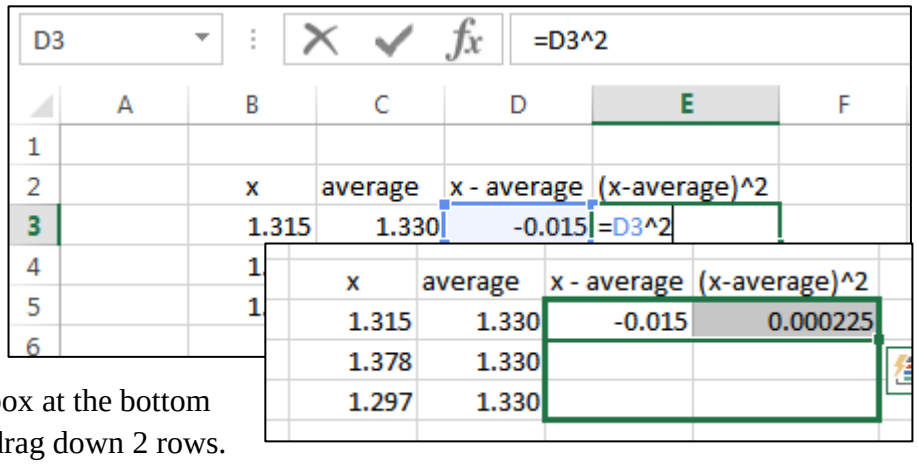
Finding Standard Deviation with Excel

Set up your Excel sheet like table shown below, with the numbers in a column, and the average in the column next to them. The labels are just to make things easier for you.

Select the next cell in your first row (here in my example D3), then type an equals sign, =, click on the first number (here B3), then type a minus sign, -, then click the average next to it (here C3), then press ENTER.

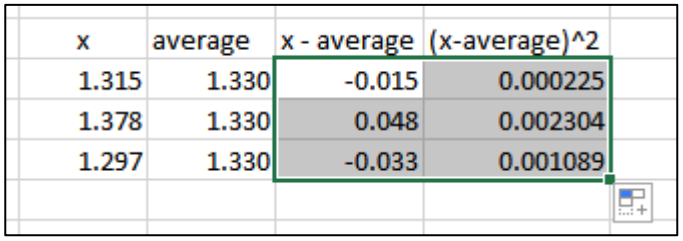
Then select the next cell (here E3) and type an equals sign, =, and click the previous cell (here D3), then type ^2, press ENTER. This will square the value for the value in that cell.

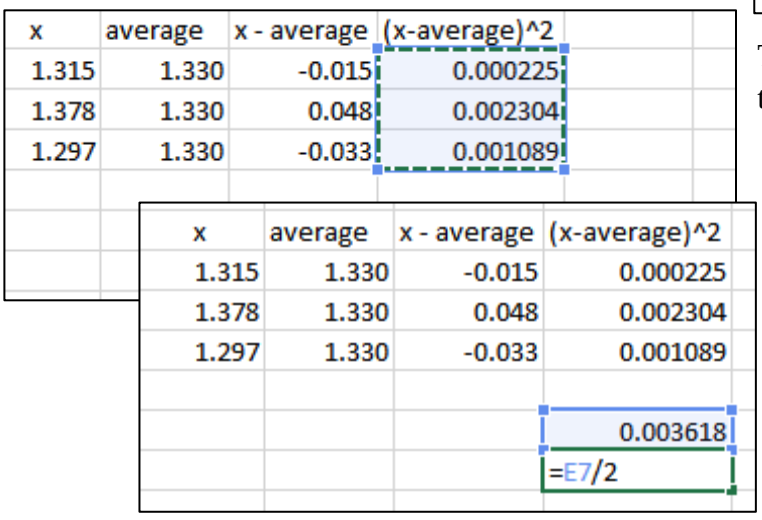


Then click and drag to select cells D3 and E3. While selected, click the little box at the bottom right-hand corner of the selection, then drag down 2 rows. This will copy the formulas to the other cells.

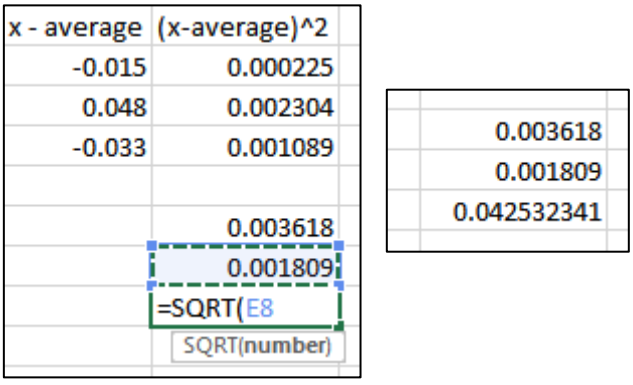
Then find the sum of the cells by selecting a blank cell, then typing: =sum, select the sum function from the dropdown box, then click and drag to select the 3 cells. Press ENTER.

This value is $\Sigma(x_i - \bar{x})^2$





Then you can divide this number by n-1, then find the square root by using the SQRT function.



Note: If you use the standard deviation function in Excel, =STDEV.P(value1,value2,…). Excel will give a value smaller than the value that is calculated from the procedure above.

The reason is the program divides by n, instead of (n-1); the formula that we use.