Ye Olde Measurement of Equivalent Weight Via Weak Acid/Strong Base Titration

I. Introduction

One of the most important relationships established in the early days of chemistry was that between the weight of a given sample and the number of molecules contained in that sample. The chemist thus always learns in chemistry school to be very good at weighing samples: with a stellar (and very expensive!) analytical balance, he/she can measure the mass of a reasonable size sample to five or six significant figures.

This is all well and good. But in the vast majority of situations, the chemist needs to know much more than just the mass. The chemist needs to know the *precise number* of molecules contained in the sample--at least to three significant figures. Only then can informed judgments be rendered regarding the amount of sample needed for a particular application. It is not an overstatement to say that virtually all of chemistry research at the lab bench begins with relating the mass of a sample of interest to the number of molecules contained in that sample. 30.00 grams of benzoic acid corresponds to ??? number of molecules. 1.25 milligrams of Ribonuclease A corresponds to ??? number of protein molecules. 5.28 tons of carbon dioxide corresponds to ???? number of molecules ejected into the atmosphere by buses, trains, and automobiles. There are chemists and policy makers out there who really need to know these things right now.

Now it turns out that the relationship between mass and numbers of molecules is not as cut-and-dry as might be expected. There is **molecular mass**; we all know about this via high school chem and Chem 101. In turn, we all know how to compute mass values based on molecular formulae: $C_7H_8O_2$, $C_{21}H_{45}O_2$, $C_{12}H_{12}O_6$, and so forth. Such calculated values correspond to the *averages* of mass quantities which can be measured experimentally using a mass spectrometer. The word "averages" is critical here because the spectrometer is sensitive to the mass differences posed by atomic isotopes: carbon-13 versus carbon-12, hydrogen-2 versus hydrogen-1, and so on.

Then there is the concept of **equivalent mass**. This flavor of mass is important where the molecules investigated by the chemist are subject to a reaction in the laboratory.

Important: the equivalent mass of a molecule is the number of grams of the molecule that react with 1.00 mole of a known reagent.

Let's imagine that the chemist is interested in Molecule A with a molecular mass of 120 grams per mole. Let's imagine further that Molecule A undergoes the following reaction with Reagent Molecule B:

$$1A + 2B = 1C$$

Clearly 1.00 mole of A-molecules die in the reaction for every 2.00 moles of B-molecules that die. The stoichiometry also informs the chemist that 0.50 moles of A-molecules die in the reaction for every 1.00 moles of B-molecules that die.

Important: The chemist would state that equivalent mass of Molecule A reacting with Reagent Molecule B is 60 grams per mole. It takes 60 grams of Molecule A to die for every 1.00 mole of B that dies via the chemistry.

Let's try again with another scenario. Say that the chemist is interested again in Molecule A with a molecular weight of 120 grams per mole. Let Molecule A undergo the following reaction with Reagent Molecule D:

$$1A + 1D = 1E$$

Clearly 1.00 mole of A-molecules dies for every 1.00 mole of D-molecules that die in the reaction. In mass terms, 120 grams of Molecule A are consumed for every 1.00 mole of D.

Still Important: the equivalent mass of Molecule A reacting with Reagent Molecule D would interpreted as 120 grams per mole. It takes 120 grams of Molecule A to die for every 1.00 mole of D that dies.

We arrive at yet another important point:

The equivalent mass of a molecule *can be* different from the formula mass in some cases. But it can also be the same in still other cases. This is a confusing but true facet of the relationship between sample mass and number of molecules contained. The equivalent mass of Molecule A, unlike formula mass, depends on more than just Molecule A taken by itself. Equivalent mass also depends critically on the reaction used by the chemist to study Molecule A.

Formula mass, equivalent mass, and reaction stoichiometry are center-stage concepts in chemistry. The purpose of this Chem 112 experiment is to gain a deeper understanding of these concepts. To that end, one will gain hands-on experience with a handful of organic weak acids plus titrations with strong acids and strong bases. One will use titration methods to (1) identify the concentration of a diluted strong base solution, (2) measure the equivalent mass of an unknown organic acid compound and, (3) identify the molecular identity of the unknown acid.

II. The Reactions Between Weak Organic Acids and Strong Bases

Many--but certainly not all--organic weak acids feature a **carboxyl functional group**. The abbreviated Lewis structures of several organic acids are illustrated on the following page.

Importantly, it is the **hydrogen atom contained in the intact carboxyl group** that confers the acid behavior of the molecule. An intact carboxyl group is said by the chemist to pose one acidic hydrogen atom.

You probably see where the discussion is going. An organic acid molecule **can contain more than one carboxyl group**. In turn, an organic acid molecule can feature **more than one acidic hydrogen atom**. Organic acid molecules are tricky objects given that they contain several hydrogen atoms--not all of which are acidic in nature. The non-acidic hydrogens really do

nothing in acid/base reactions except go along for the ride.

Important: For the weak acids studied in this Chem 112 experiment, only the hydrogen atoms affiliated with carboxyl groups will behave as acidic hydrogens. If a chemist needs to count the number of acid hydrogens in an organic sample from this lab period, he/she will begin by counting the number of carboxyl groups.

The next critical idea is one driven home in the acid/base chapters of Chem 102:

Acid/Base chemistry revolves around hydrogen and hydroxide ions in the aqueous phase: $H^+(aq)$ and $OH^-(aq)$. These parties combine chemically in a 1:1 ratio to make liquid water molecules:

$$1\ H^+(aq) + 1\ OH^-(aq) = 1\ H_2O(liq)$$

As one learns, the equilibrium of the above famous reaction is **very heavily weighted on the right-hand-side**: $K \approx 10^{+14}$ at temperature 298 K.

The main activity of this experiment will be to measure the number of moles of hydroxide ions required to neutralize a **measured amount of organic acid**. The results of this measurement will yield the equivalent mass of the acid.

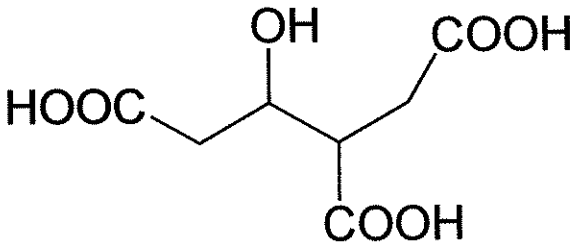
How will the chemist know when an acid is neutralized? The chemist will use an age-old but very effective trick of acid/base titrations. The chemist will specifically dope each acid sample with a tiny amount of phenolphthalein. This molecule is itself a weak acid material. Its claim to fame is that it is colorless under acidic conditions and a bright shining pink under basic conditions. The chemist will add hydroxide ions to the sample of interest quantitatively--this is the famous process known as **titration**. The chemist will know that the acid sample has been neutralized when the pink color conditions of the dopant ever so slightly persist.

III. Experimental and Calculations

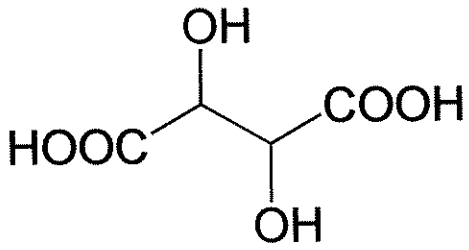
A. Getting Started

Please put on both safety glasses and latex gloves. Every team should then organize the following equipment:

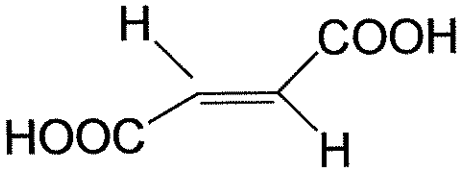
Erlenmeyer flasks of volume ≥ 250 milliliters
watch glasses
small graduated cylinder
large graduated cylinder
beaker with volume ≥ 100 milliliters
2 burets with stopcocks



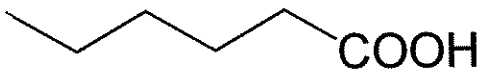
Citric Acid



Tartaric Acid



Maleic Acid



Hexanoic Acid

rubber stoppers for Erlenmeyers

As per usual, each team member should apportion five to seven pages of the lab notebook to record all data for the experiment.

Now use a clean small graduated cylinder to collect 7.0 milliliters of 6.00 molar aqueous sodium hydroxide. Collect the sample in the hood--latex gloves must be worn during this step.

!!!Note that the solution used here is basically concentrated Drano = Liquid Plumber used to unclog kitchen and bathroom drains. Please be extremely careful!!!!

Now add the 7.00 milliliters of 6.00 molar aqueous sodium hydroxide (Drano) to a clean Erlenmeyer flask. Slowly add 200 milliliters of distilled water. Mix the sample slowly, carefully, and thoroughly. Then seal the sample using a rubber stopper.

At this juncture, please calculate and record the approximate concentration of the diluted sodium hydroxide solution in moles per liter.

Now collect 75 milliliters of the HCl solution stored in the hood using the larger graduated cylinder. Pour this liquid carefully into a beaker and then cover the sample using a watch glass. The concentration of the HCl is important to this experiment; please record its value at this time.

!!!The HCl is a strong acid solution. Please exercise every caution handling this solution!!!

Now carefully but thoroughly rinse two burets needed for this experiment with water from the faucet. Let the burets drain as much as possible, then plug in the stopcocks. Turn the stopcocks to the "open" position. The better the drainage, the better the experimental results.

After a few minutes of drainage, please rinse **one of the burets** with a few milliliters of HCl solution. One needs less than five milliliters for this operation, but the rinsing--and further drainage--has to be thorough.

Now close the stopcock. Mount the buret in the upright position. Add 3.00 milliliters of HCl solution to the buret.

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There is the second buret to attend to, having been washed thoroughly. After it has drained, then rinse **this buret** with a few milliliters of **dilute NaOH solution**.

!!!Absolutely, positively, do not use the concentrated NaOH for this task!!!

As with the HCl rinsing, one needs less than five milliliters of base solution for this task. And the follow-up rinsing--and further drainage--has to be thorough.

Now plug in and close the stopcock on the second buret. Mount the buret tube in the upright position. Add 3.00 milliliters of **dilute NaOH solution** to the buret. Again, stay clear of the concentrated NaOH.

Label the burets "HCl" and "NaOH". We are now ready to rumble.

B. The Next Steps

Add 50 milliliters--close enough is good enough--of distilled water to a clean Erlenmeyer flask. Add 25.0 milliliters of the HCl solution to the water from the HCl-labeled-buret.

There will be some phenolphthalein indicator samples stored in the hoods. Add add three or so drops of indicator solution to your dilute acid solution in the Erlenmeyer flask.

Now begin titrating the acid sample in the Erlenmeyer flask **with the NaOH solution**. Add the NaOH solution *quantitatively* to the acid solution in small increments while swirling and shaking the acid solution.

Important: with each incremental addition, one should see a flash of pink color. The color should appear momentarily at the site of reaction and then vanish as the flask is shaken. The disappearance of the color will occur faster than not when the sample is far from the titration endpoint. The disappearance will transpire more slowly as the endpoint is approached.

Also Important: Please aim for the titration point at which the pink color appears and then ever so slightly persists. This is the endpoint of the titration.

Once the endpoint has been identified....

(i) Calculate the number of moles of hydrogen ions in the 25.0 milliliters that went originally into the Erlenmeyer flask.

This number of moles of hydrogen ion is equivalent to the number of moles of hydroxide ion that were needed to neutralize the solution.

(ii) Then calculate the concentration of sodium hydroxide solution. Here we divide the number of moles of hydroxide ion from (i) by the number of liters added from the NaOH buret to the Erlenmeyer flask.

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Now go back to the beginning of **Section IIIB**. Perform a titration of a **20.0 milliliter sample of HCl** from the HCl buret to which **40.0 milliliters of distilled water** have been added. Repeat all the steps in order to arrive at a second value for the concentration of hydroxide ions in the

NaOH buret.

Compute and record the **average concentration** of hydroxide ions in moles per liter.

C. Titrating the Organic Acid with NaOH solution

The lab assistant will provide each team with an organic acid sample. Please record the number of the sample in the lab notebook. The sample material should be fairly benign. Nonetheless, please wear latex gloves at all times.

Divide the sample into two more-or-less equal portions. Weigh each portion as accurately as possible. Add each portion to a separate Erlenmeyer flask. Add 50.0 milliliters of distilled water to each flask. Swirl each mixture until all of the solid material dissolves. If solubility problems plague this step of the lab experiment, add 5 - 10 milliliters of ethanol to the sample. A little bit goes a long way. This should fix matters--hopefully!

To each organic acid solution add a few drops of phenolphthalein indicator.

Now add more of the diluted NaOH solution to the NaOH buret. At this juncture of the lab experiment, each team should be fairly confident of knowing the concentration of this NaOH solution.

Now start titrating the **weak acid sample** in the Erlenmeyer flask using the NaOH solution. As with titrating HCl, add the NaOH solution from the buret in small increments while swirling and shaking the receiving flask.

We know what to expect: with each incremental addition, there should be a flash of pink color. The color should vanish, however, as the flask is shaken. The disappearance of the pink color will be relatively fast when one is far from the titration endpoint. The disappearance will occur more slowly as the endpoint is approached.

Aim for the titration point at which the pink color appears and then ever so slightly persists. This is the endpoint of the titration.

Then...

(i) Calculate the number of moles of hydroxide ions that were transferred from the NaOH buret to the receiving flask. For this task, use the **average concentration of OH^-(aq)** from **Section IIIC**. The final result represents the number of moles of hydroxide ion that were needed to neutralize the weak organic acid.

(ii) Calculate the equivalent mass of the acid by solving the following proportion:

$$\frac{\text{number of moles of } OH^-(aq) \text{ from buret}}{\text{mass of acid sample in grams}} = \frac{1.00 \text{ mole of } OH^-(aq)}{\text{??equiv mass of acid??}}$$

Now repeat the titration steps on the second weak acid sample. Determine the equivalent mass a second time. Compute the average value of the equivalent mass.

D. Identifying the Organic Acid

There are four possible identities for the organic acid sample:

Citric Acid
Tartaric Acid
Maleic Acid
Hexanoic Acid

The abbreviated Lewis Structures of these compounds were illustrated in **Section II**. Each team should use their experimental data to identify the acid material. If the smell of an open bottle does not drive people to run from the lab, the sample is not hexanoic acid.

IV. Preliminary Report

Please complete the preliminary report sheet found on the last page of this handout. Each lab partner should complete his/her own sheet based on the team data. Turn in the preliminary report to the lab assistant before leaving.

V. Final Report

Please write a final report based on data recorded for this experiment. Each partner of a team must write and submit his/her own lab report. Each report must be typewritten and should adhere to the following outline.

A. Introduction: Please describe the purpose of the experiment in your own words. Please explain the concept of equivalent mass. Describe the type of chemical reaction that is used in this experiment to investigate and measure equivalent mass.

B. Experimental: Please summarize the experimental method. Please note any deviations from the procedure described in the handout.

C. Results: Please clearly present the results of **four titration experiments**. A table will assist in organizing the following interrelated quantities:

titrating reagent concentration
milliliters of buret reagent added
moles of buret reagent added
moles of receiver flask material
concentration of receiver flask material

Please detail how the equivalent mass of the unknown acid sample was computed. Please describe the basis for which the unknown acid sample was identified.

D. Discussion: Please discuss the significance of experimental results in your own words. What happened in the experiment that was expected? What--if anything--happened that was not anticipated?

The titration endpoints were identified on the basis of color changes. Please note ambiguities in the experimental results.

Please describe the next experiment that you would undertake in an investigation of the equivalent mass of the weak organic acid assigned to your team.

Ye Olde Pre-Lab Assignment

1. Please calculate the molecular mass affiliated with each Lewis structure of **Section II**.

2. Let each molecule illustrated in Section II be subject to neutralization with sodium hydroxide. Please calculate the equivalent mass for each compound.

3. A chemist combines 7.00 milliliters of 6.0 molar sodium hydroxide with 200 milliliters of distilled water. What is the new concentration of hydroxide ions?

4. The chemist uses the diluted solution of (3) to titrate 24.50 milliliters of 0.1005 molar HCl. How many milliliters of the base solution are required to reach the equivalence point?

Preliminary Report

Name:

Lab Partner's Name:

Identification Number of Weak Acid Sample:

Lab Assistant's Name

1. [$OH^-(aq)$] determined from the first titration:

2. [$OH^-(aq)$] determined from the second titration:

3. Equivalent mass of acid determined from the third titration:

4. Equivalent mass of acid determined from the fourth titration:

5. Likely identity of the organic acid sample: