Introductory Chemistry Lab: Titration

Outcomes

As a result of today's laboratory, you will have:

Used burets to measure the volumes of solutions.
Standardized a NaOH solution by titrating against the primary standard HCl.
Titrated acetic acid with standardized NaOH.

Prelab

Prepare a Title (can use the lab handout for this), Purpose (a concise statement) and a Procedure (short "to do" list … see "Writing a Procedure" in the lab handouts folder), and Data Tables.

Purpose

To determine the molarity of an acetic acid solution.

Background Information

Vinegar is a dilute solution of acetic acid (CH_3COOH, HOAc, or $HC_2H_3O_2$) in water. The acetic acid will undergo a neutralization reaction with a base such as sodium hydroxide (NaOH).

$$\mathbf{NaOH_{(aq)} + HC_2H_3O_{2\,(aq)} \rightarrow NaC_2H_3O_{2\,(aq)} + HOH_{(l)}}$$

By adding just enough NaOH to consume all of the acetic acid, the amount of acetic acid can be determined.

Titration is the process of using a known concentration (the standard solution) to determine the quantity (molarity) of another solution. The key part of the titration process is the determination of the point when just enough of each solution has been added to complete the reaction. This point is known as the "equivalence point" or "end point." While there are many ways to solve the problem of finding the equivalence point, the most common technique is to use a small amount of an "indicator" (a substance that changes color at or near the equivalence point). The point in the titration where the indicator signals that the reaction is complete by changing color is called the "endpoint."

Once the endpoint has been determined and the number of milliliters of NaOH and of acetic acid reacted are known, the number of moles of NaOH added can be calculated from its volume and molarity. The number of moles of acetic acid that reacted can be calculated using the coefficients of the balanced equation for the neutralization reaction. The molarity of the acetic acid can be calculated from the number of moles of acetic acid present in its known volume in liters.

The calculations require that the concentration of NaOH be accurately known. However, when a typical NaOH solution is prepared its concentration is not well known. There are two reasons for this. One is that solid NaOH readily absorbs moisture from the air. This means that as NaOH is being weighed on the balance its mass is increasing due to absorbed water. The second problem is carbon dioxide gas present in the air and will react with the NaOH.

$$CO_{2\,(g)} + H_2O_{(l)} \rightarrow H_2CO_{3\,(aq)}$$

$$\downarrow$$

$$H_2CO_{3\,(aq)} + NaOH_{(aq)} \rightarrow Na_2CO_{3\,(aq)} + H_2O_{(l)}$$

To overcome this problem, the concentration of the NaOH solution is first determined by titrating the NaOH against a known acid, HCl, concentration. This process is called "standardization."

$$\mathbf{HCl_{(aq)} + NaOH_{(aq)} \rightarrow HOH_{(l)} + NaCl_{(aq)}}$$

The volumes of HCl and NaOH that completely neutralize each other in the standardization are measured. Knowing the concentration of the standard allows calculation of the concentration of unknown.

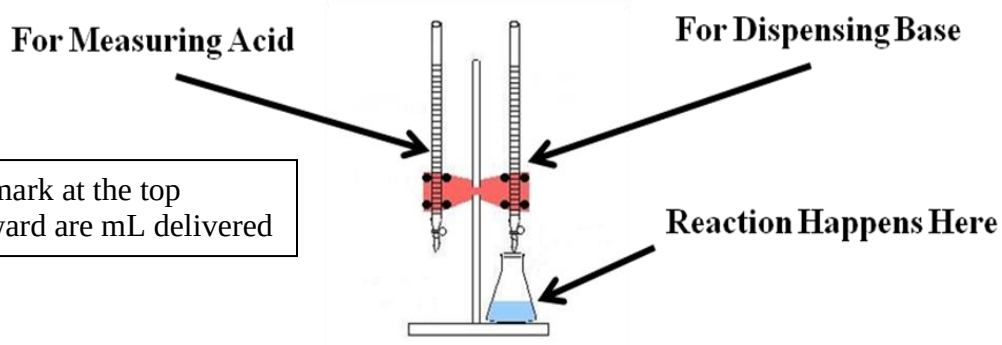
Warning: Wear safety goggles. NaOH and HCl are strong chemicals that are used as drain cleaners.

Procedure Work in pairs

Part I. Standardization of NaOH Solution

1. Scrub the inside of one 250-mL Erlenmeyer flask and three 150-mL beakers with soap solution. Do not put soap into the burets. Then rinse the glassware including your two burets thoroughly with deionized water. Make sure that a little of each rinse is allowed to run through the tip of the buret by opening the stopcock. Dry the burets and the flask on the outside with paper towels and then let them drain "dry" on the inside. Dry the inside and outside of the beakers thoroughly with paper towels.
2. Label four beakers HCl, NaOH, Acetic Acid, and Waste.
3. The instructor will give you the HCl concentration. Record this value in Table 1 as Molarity HCl..
4. Put ~75 mL of HCl in a beaker labeled HCl
5. Put ~75 mL of NaOH in a beaker labeled NaOH.
6. Label one buret "HCl" and rinse the inside of this buret with about 5 mL of HCl. Be sure to run some of the HCl rinse through the tip of the buret. Repeat this HCl rinse two more times. Your goal during the three rinses is to rinse out the deionized water on the inside of the buret and replace it with HCl. It is important that the HCl used in the titration not be diluted with water from wet burets.
7. Label the second buret "NaOH" and rinse out the water from this wet buret with three 5 mL portions of NaOH like you did in the step above with the HCl buret.
8. Be sure that the burets do not leak around the stopcock. If you suspect a leak, ask your instructor to help you tighten the stopcock. Then attach a double buret clamp to a ring stand and place one buret in each side of the clamp with the tip end down. Be sure the burets are clamped securely in both rubber gripping "teeth" of the clamp and are vertical (not cocked to one side).
9. Fill each buret with the appropriate solution. Then fill the tip of each buret by opening the stopcock and allowing some of the solution run through it into the waste beaker. The buret is calibrated "to deliver" and so a small amount of liquid at the very end of the tip is acceptable.
10. Fill each buret to the 0.00 mark. Record the initial buret (two decimal places) reading for the HCl solution and for the NaOH solution in Table 1.
11. **Using the buret**, add ~20 mL of HCl to the Erlynmeyer flask.
12. Add two drops of phenolphthalein indicator solution to the Erlenmeyer flask. **Do not forget the indicator.** Put a white paper sheet under the flask to make seeing faint color changes easier**.**

The Titration Assembly

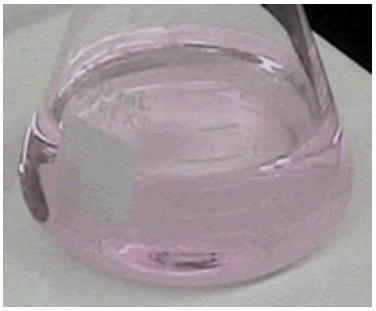


Burets have the 0.00 mark at the top
Measurements downward are mL delivered

13. Titrate the hydrochloric acid in the flask by placing it under the "NaOH" buret and adding sodium hydroxide to the flask. While you are adding the sodium hydroxide continuously mix the NaOH and HCl by swirling the contents of the flask. As more NaOH is added the solution in the flask will remain pink for a longer time before it clears upon complete mixing. When the pink persists (hangs around) longer, this is a sign that you are getting close to the endpoint of the titration. Close the stopcock to stop adding NaOH.

14. Rinse down the sides of the flask and the tip of the buret with deionized water. This is to make sure that all the NaOH from the buret has been mixed (and reacted) with the HCl. Note that adding water to the flask does not change the volume of HCl or NaOH added from the burets and will not affect your results.

15. If the contents of the flask are still colorless at this point, add NaOH dropwise until the addition of just one drop causes the solution in the flask to remain a faint pink. The endpoint in the titration occurs when just one drop, or even just a half-drop, changes the color of the flask contents to a very faint pink.



Correct End Point

Too Much Base

16. If the solution in the flask is a darker pink color, you have added too much NaOH. Go back to the HCl buret and add acid to the flask drop-wise until the color just disappears. Be sure to mix the contents of the flask well and to rinse down the sides of the flask and tip of the buret with deionized water.

17. Now return to the NaOH buret and add one drop (or a half-drop) of base to the flask. Rinse down the sides of the flask and buret tip. The addition of one drop should turn the solution faint pink. Continue going between the HCl and NaOH until just one drop of added base changes the color from colorless to faint pink.

18. Record the final buret volume readings for the HCl and for the NaOH in Table 1.

Part II. Titration of Acetic Acid

1. Put ~75 mL of acetic in the beaker labeled "Acetic Acid."
2. Empty and rinse the Erlenmeyer flask three times with deionized water. Empty the HCl buret and re-label it Acetic Acid. Rinse the Acetic Acid buret, including the tip, with deionized water several times. Then, as before, rinse out the water inside the buret and its tip with three 5 mL portions of acetic acid. Then fill the buret with acetic acid.
3. Fill up the Acetic Acid buret close to 0.00. Record the initial buret volume for the acetic acid in Table 1.
4. Refill the "NaOH" buret from the "NaOH" beaker. Record the initial buret volume reading in Table 1.
5. **Using the buret**, add about 20 mL of acetic acid to the Erlenmeyer flask.
6. Repeat the titration process as in steps 12 – 15 of Part 1 using acetic acid in place of HCl.

Clean Up

1. When finished, dispose of all chemicals in the sink with lots of water.
2. Remove the labels from all glassware.
3. Rinse all glassware several times with tap water and then several times with deionized water.
4. Towel dry all glassware and put away.
5. Return all equipment to the proper storage place.

Data

Table 1: Buret Measurements

Standardization of NaOH			
Molarity of HCl (from instructor):			
Final HCl buret reading (mL)		Final NaOH buret reading (mL)	
Initial HCl buret reading (mL)		Initial NaOH buret reading (mL)	
Titration of Acetic Acid			
Final HOAc buret reading (mL)		Final NaOH buret reading (mL)	
Initial HOAc buret reading (mL)		Initial NaOH buret reading (mL)	

Calculations ***Show all calculations including units with the appropriate number of sig figs.***

Calculate the molarity of the standardized sodium hydroxide (NaOH).
Calculate the molarity of the acetic acid (HOAc) solution.
Record results in Table 2.

Results

Table 2: Summary

Standardization of Sodium Hydroxide (NaOH)		**Titration of Acetic Acid (HOAc or CH_3COOH))**	
Molarity NaOH (mol/L)		Molarity HOAc (mol/L)	

Conclusion

Summarize your results (list molarities of NaOH standard and acetic acid).

Questions

1. Vinegar is a solution of acetic acid (the solute) in water (the solvent) with a solution density of 1010 g/L. If vinegar is 0.80 M acetic acid, what is the % by mass concentration of acetic acid in vinegar?

2. How would each of the following errors affect the experimental value of your molarity of acetic acif? Would your Molarity value be too high, too low, or unaffected? Explain your answer.

a. if you titrated your acid sample to a bright pink rather than faint pink endpoint?

b. if your "acetic acid" buret was still wet inside with deionized water when you filled it with acetic acid?

c. if you recorded the final volume of NaOH as "39.88 mL" instead of "38.88 mL"?