Acid / Base Titrations

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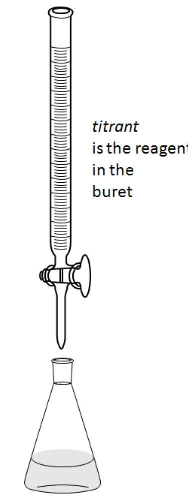
Objectives: Determine the concentration of a base solution using an acid standard.
Optional: Precipitate an ionic salt for percent yield determination using the standardized base or determine the concentration of an acid using the standardized base.



Background: Carefully Read Sections 4.4 and 4.8 in Tro 2nd Edition
The most common method for determining the concentration of a solution is via titration. A titration involves reacting a solution of unknown concentration against a known **standard** to reach an **endpoint**, often signaled by an abrupt color change in the reaction mixture. A **standard** is a compound whose composition is unchanged by light or heat and one that is not **hygroscopic,** meaning that it does not readily absorb water from the atmosphere. The titrant reagent is carefully added to the other reagent until an **equivalence point** is reached (p 158), the point where stoichiometric amounts of the reactants have combined and both are limiting. The **endpoint** of a titration typically occurs after a few drops of excess titrant are added, past the equivalence point. The majority of acid / base reactions involve colorless products and therefore the equivalence point and endpoint are not visually detectable. To allow for the identification of the endpoint, an **indicator** is added at the start of the reaction. An **indicator** (p 158) is a substance that changes color depending on the **pH** of the solution. **pH** is indicative of the amount of hydronium, H_3O^+, in solution.

In this lab you will be standardizing (determining the concentration of) a sodium hydroxide solution by titrating it against an oxalic acid dihydrate standard according to the reaction:

$$H_2C_2O_4 \cdot 2H_2O\ (aq) + 2\ NaOH\ (aq) \rightarrow Na_2C_2O_4\ (aq) + 4\ H_2O\ (l)$$

You will begin by weighing an amount of oxalic acid dihydrate and dissolving it in water. The amount of water need only be approximate as the amount of hydronium in solution will be determined by the mass of oxalic acid. To this solution you will add the indicator phenolphthalein. Phenolphthalein is colorless in the presence of excess hydronium but turns pink in the presence of excess hydroxide. Sodium hydroxide of approximately 0.1 M concentration will be the titrant added to the acid until your reach the endpoint. The mass of oxalic acid dihydrate, the volume of sodium hydroxide used, and the stoichiometric relationship of these two compounds will enable you to determine the concentration of the sodium hydroxide to an increased number of significant figures.

Once you have determined the concentration of the sodium hydroxide solution, then you can use that sodium hydroxide for other tests. Your instructor might provide you with an additional procedure to perform additional titrations.

Safety:
Wear safety goggles. Wash your hands thoroughly before leaving the lab.
Sodium hydroxide, Oxalic Acid Dihydrate, and Phenolphthalein

- If ingested, immediately rinse mouth and drink plenty of water (200-300 mL)
- If it comes in contact with your skin, remove contaminated clothing and wash before reuse. Wash the skin immediately with soap and water.
- If it comes in contact with your eyes, rinse eyes for at least 15 minutes with water. Remove contact lenses if worn prior to rinsing.

Disposal:
Dispose of mixture in Erlenmeyer flasks down the drain.
Dispose of excess NaOH down the drain with copious amounts of water. This is acceptable because the concentration is much less concentrated than that of Liquid Plumber Drain Cleaner.

Procedure

I. Preparation of Oxalic Acid Dihydrate Solutions for Titration

1. Thoroughly wash 3 Erlenmeyer flasks with soap and tap water.
2. Rinse the flasks first using tap water a minimum of 3 times each.
3. Rinse the flasks second with distilled water a minimum of 3 times each.
4. Place a piece of weigh paper on the balance and hit the tare/zero button.
5. Using weigh paper, measure out a sample of oxalic acid dihydrate, approximately 0.2 – 0.25 g in mass.
6. Record the exact mass on the data sheet to the nearest 0.001 g.
7. Add the sample of oxalic acid dihydrate to the first Erlenmeyer flask and label that flask #1. Carefully rinse remaining residue from the weigh paper using distilled water from a water bottle.
8. Add approximately 50 mL of distilled water to the Erlenmeyer flask.
9. Mix until all of the oxalic acid dihydrate has dissolved.
10. Add 2 drops phenolphthalein to the Erlenmeyer flask.
11. Repeat steps 4-10 for the other two Erlenmeyer flasks, labeling the flasks #2 and #3 accordingly.

II. Preparing the NaOH Solution for Titration

1. Thoroughly wash a buret and rinse it using distilled water.
2. Close the stopcock.
3. Add approximately 5 mL of NaOH to the buret.
4. Gently roll the buret back and forth to coat the sides of the buret.
5. Drain the rinse solution through the buret tip into a discard container.
6. Repeat steps 3-5.
7. Close the stopcock and add approximately 50 mL of NaOH solution to the buret.
8. Drain a small amount of NaOH solution through the buret tip into the discard container to remove all bubbles.
9. Record this volume of NaOH as your initial buret reading for the first experiment.

III. Standardizing the NaOH Solution

1. Place Erlenmeyer flask #1 containing the oxalic acid dihydrate under the buret and lower the buret so that the buret tip is below the mouth of the flask. **(see Figure 3.7 on page 90 of your text)**
2. Add NaOH solution to the Erlenmeyer flask by swirling the flask with one hand and controlling the stopcock with the other.
3. As you approach the endpoint take particular care to add the NaOH solution dropwise.
4. Add NaOH solution to the flask until a pale pink color is obtained after one drop and persists for 30 seconds.
5. Have your instructor approve your result.
6. Record this volume as your final buret reading for the first experiment.
7. Discard the solution in the Erlenmeyer flask down the drain.
8. Refill the buret, using a funnel, to approximately 50 mL of NaOH. Record this volume as the initial reading for the 2nd experiment.
9. Repeat steps 1-8 for the other two Erlenmeyer flasks using flask #2 and #3 accordingly.

Acid / Base Titrations: Pre-Laboratory Assignment

1. Give at least two characteristics desirable in a primary standard.

2. Define *equivalence point* and *titration end point*. Do these two points in a titration occur when the same volume of titrant has been added? Explain.

3. A student standardized a NaOH solution using $KHC_8H_4O_4$. Three different $KHC_8H_4O_4$ samples were dissolved in water and titrated with the same NaOH solution, each to a phenolphthalein end point. The pertinent reaction is:

$$KHC_8H_4O_4(aq) + NaOH(aq) \rightarrow NaKC_8H_4O_4(aq) + H_2O(l)$$

Results from these three runs are summarized in the data table below. Complete the following summary table of calculated values using the given experimental measurements.

	1	2	3
Mass of $KHC_8H_4O_4$ titrated, g	0.452	0.470	0.442
Volume of NaOH(aq) required, mL	15.45	15.99	15.05
Number of moles of $KHC_8H_4O_4$ titrated, mol	_________	_________	_________
Number of moles of NaOH required, mol	_________	_________	_________
Volume of NaOH solution required, L	_________	_________	_________
Molarity of NaOH solution, mol/L	_________	_________	_________
Mean molarity of NaOH solution, mol/L	_________		

Show clear work for the following:

a) Moles of $KHC_8H_4O_4$ titrated for Run #1

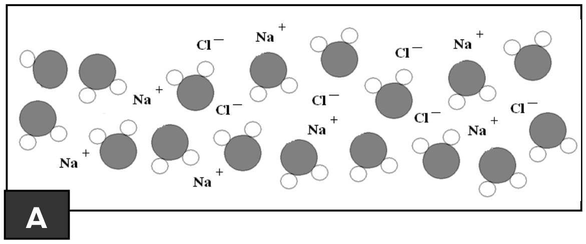
b) Moles of NaOH required for Run #1

c) Molarity of NaOH as determined by Run #1

3. Below is a set of pictorial diagrams representing five different stages of the reaction mixture during the titration of hydrochloric acid with sodium hydroxide as titrant. Water molecules, hydronium ions, chloride ions, and sodium ions in the various mixtures are shown.

a) List the diagrams by letter in the order these mixtures would appear during the titration, beginning with the earliest and ending with the final mixture.

earliest ______ _______ _______ ________ _______ final mixture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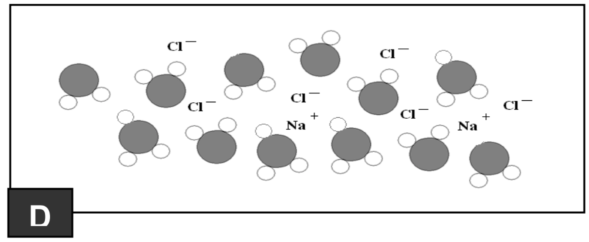
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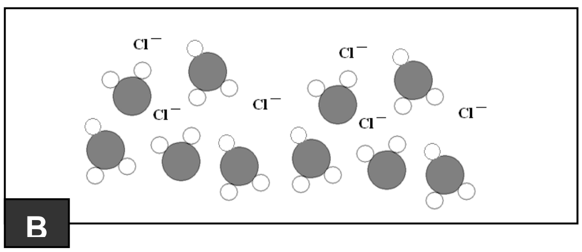
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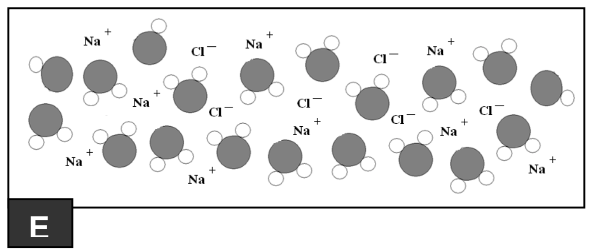
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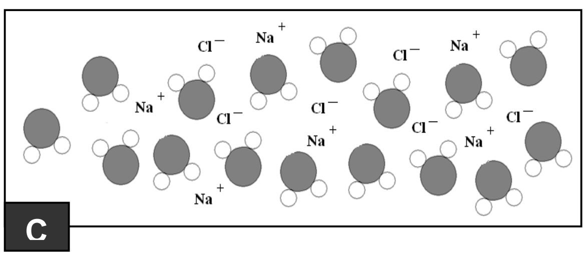
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b) Answer the following questions about the pictures above.

i) If phenolphthalein is used as the indicator, which of the solutions would be:

Colorless _________ Pink _________

ii) Which diagram represents the endpoint of the titration? ______ . Defend your choice.

iii) Which diagram represents the equivalence point of the titration? ________. Defend your choice.

Data

	1	2	3	4*
Mass of $H_2C_2O_4 \cdot 2H_2O$, g	__________	__________	__________	__________
Initial buret reading, mL	__________	__________	__________	__________
Final buret reading, mL	__________	__________	__________	__________

Results

	1	2	3	4
Moles of NaOH reacted, mol	__________	__________	__________	__________
Volume of NaOH solution added, mL	__________	__________	__________	__________
Volume of NaOH solution added, L	__________	__________	__________	__________
Molarity of NaOH solution, mol/L	__________	__________	__________	__________

Mean molarity of NaOH solution, mol/L __________

* Discuss your calculated molarities for Runs 1 – 3 with your instructor to determine if additional runs are necessary

Acid / Base Titrations: Post-Laboratory Questions

1. Sodium hydroxide solutions are best stored in tightly sealed containers to avoid exposure to atmospheric carbon dioxide.

a) Sodium hydroxide readily absorbs carbon dioxide from the atmosphere. Write the balanced chemical reaction that occurs between carbon dioxide and sodium hydroxide (Hint p. 701 of Tro).

b) Explain the effect on the concentration of your sodium hydroxide if you left it uncorked in an atmosphere where there was a significant carbon dioxide concentration.

2. Why was it acceptable to discard the final solution from your titration down the drain?

3. A student used a standardized 0.205 M NaOH solution to determine the mass percent of citric acid ($H_3C_6H_5O_7$) in a mixture of citric acid and inert potassium chloride. The pertinent reaction is

$$H_3C_6H_5O_7(aq) + 3NaOH\ (aq) \rightarrow Na_3C_6H_5O_7(aq) + 3H_2O\ (\ell)$$

Sample masses and titration data are given in the table below. Do the following calculations for each titration and enter your answers in this table.

	1	2	3
Mass of mixture titrated, g	0.356	0.478	0.420
Volume of 0.205 M NaOH(aq) required, mL	19.52	26.18	23.20
Moles of citric acid titrated, mol	__________	__________	__________
Mass of citric acid in the sample, g	__________	__________	__________
Mass percent citric acid in the sample, %	__________	__________	__________
Mean percent citric acid, %	__________		

Show clear calculations for Run #1 to receive full credit for your completed data table. This includes units and clear setup.