

Titration of Acids and Bases

Lab 4

Pages 117 - 128

Prelab Pages 122 – 123

Post lab Pages 126 - 127

Experimental Aims

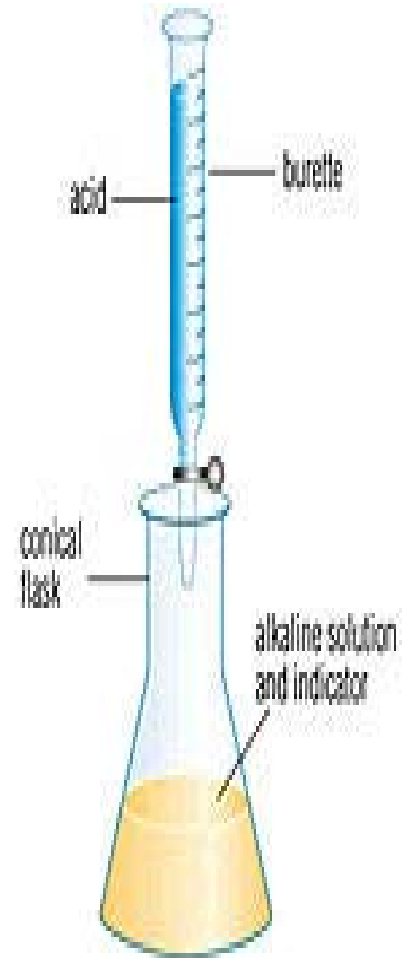
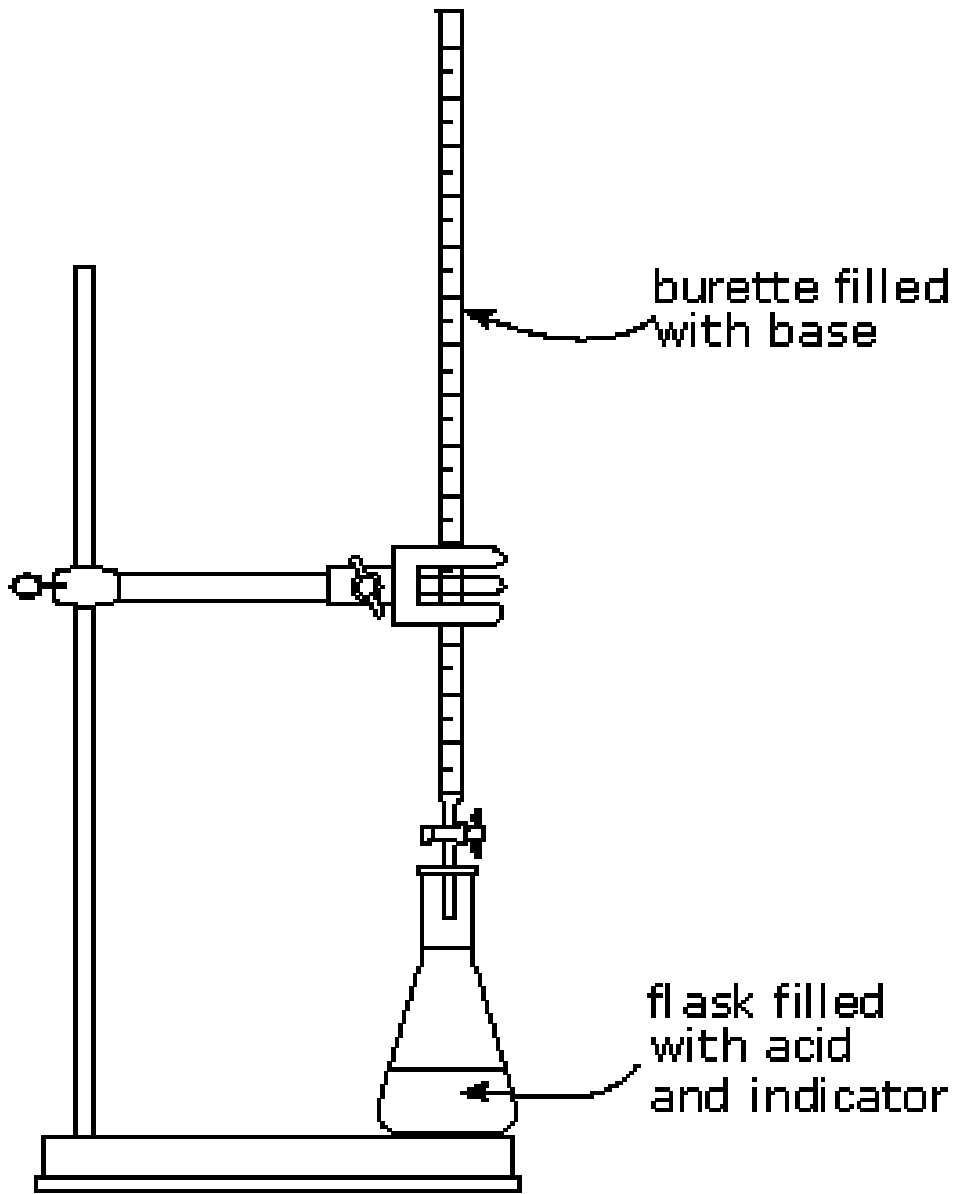
- To standardize (determine concentration) a NaOH solution
- Using your standardized NaOH calculate the molar concentration of a strong acid (HCl)
 - HOW?
 - Through titration

Techniques

- Titration
- Need reproducible results
 - 3 trials are performed (3 titrations)
- Accuracy is important

Titration

- **Titration** is a common laboratory method of quantitative/chemical analysis that can be used to determine the concentration of a known reactant.
- Volume measurements play a key role in titration, it is also known as *volumetric analysis*.



Lab Outline

- The concentration of a sodium hydroxide will be determined by titration with potassium hydrogen phthalate, ($\text{KHC}_8\text{H}_4\text{O}_4 = \text{KHP}$)
- Using the determined concentration of your sodium hydroxide solution, this will be titrated with a HCl solution to calculate its concentration.

Points to Note

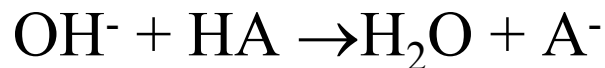
- Your solution of sodium hydroxide will be pre-made.
- Preparing a stock solution (Page 120) does not need to be performed.
- Your samples of KHP are already dried.

Introduction

- Titration is a common method of determining the amount or concentration of an unknown substance.
- The method is easy to use if the quantitative relationship between two reacting solutions is known.
- The method is particularly well-suited to acid-base and oxidation-reduction reactions.
- Titrations are routinely used in industry to analyze products to be sold. Many manufacturers are under strict standards of quality control because their products are sold for public consumption.

Background

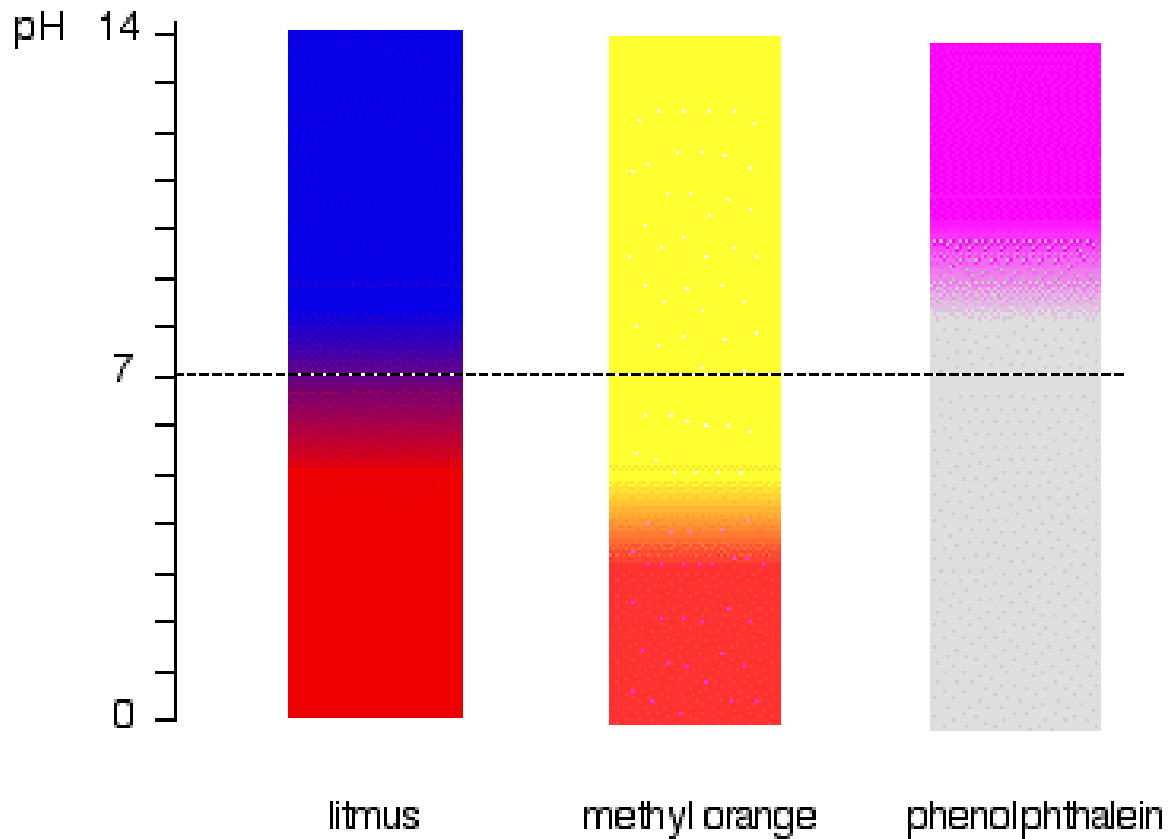
- In a titration a solution of one reactant (the titrant) is added to a measure amount of a second reactant.
- One of these reactants is called a standard (known concentration or molar mass) and the other is unknown (reactant).
- A color change (or some distinctive change) occurs when enough titrant has been added to consume all the reactant in the analyte.
- In this acid-base titration we make use of the general reaction:



What is an acid-base indicator?

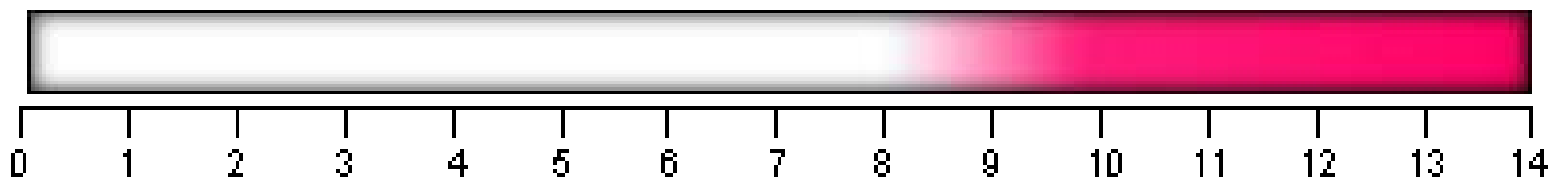
- An acid-base indicator is a weak acid or a weak base.
- The undissociated form of the indicator is a different color than the associated form of the indicator.
- An indicator does not change color from pure acid to pure alkaline at specific hydrogen ion concentration, but rather, color change occurs over a range of hydrogen ion concentrations.
- This range is termed the *color change interval*. It is expressed as a pH range.

Choice of Indicator



Phenolphthalein

Type: $\text{HIn} + \text{H}_2\text{O}$



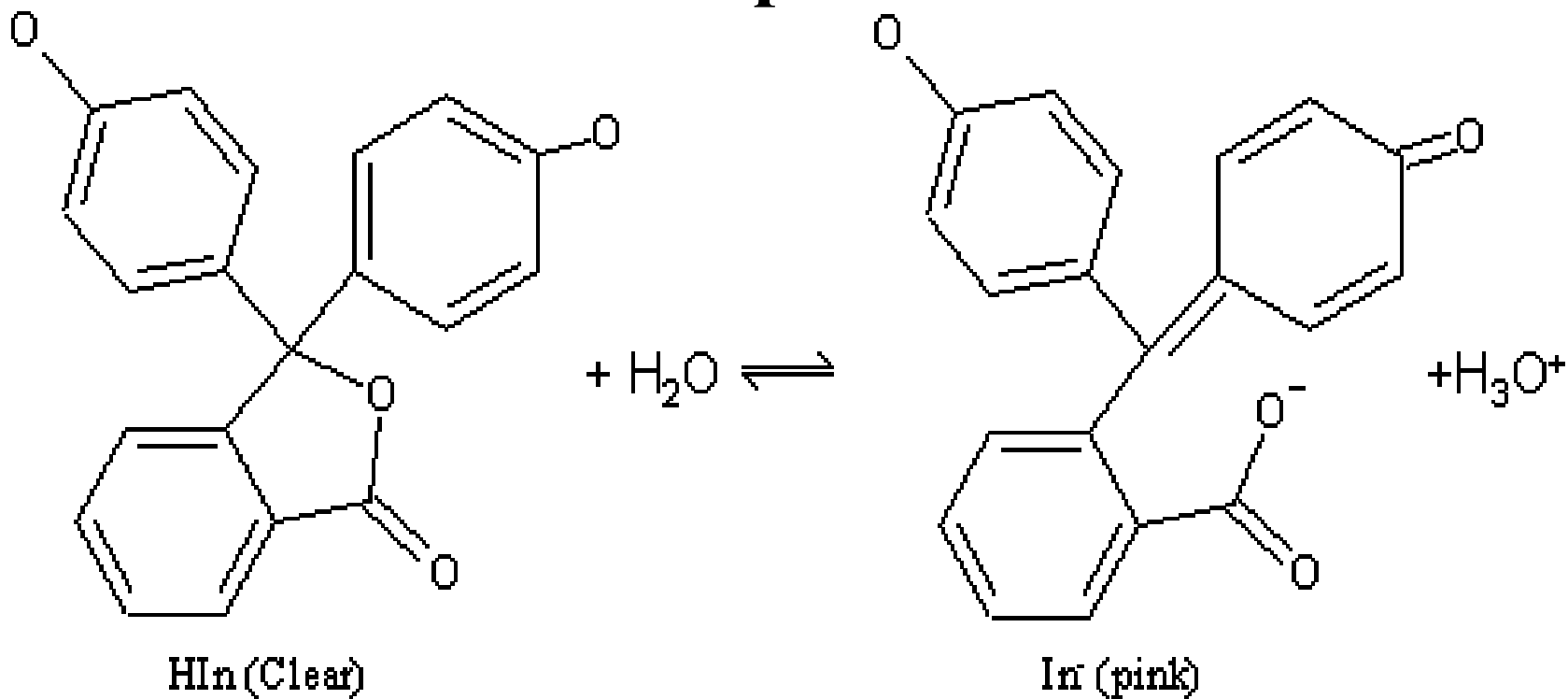
$\text{In}^- + \text{H}_3\text{O}^+$

Approximate pH range for color change: 8.0-9.8

Color of acid form: clear

Color of base form: red-violet

Phenolphthalein



The color is due to the opening up of the 5 membered ring which allows the electrons more freedom and the molecule's absorption spectrum now transmits red light.

How is an indicator used?

- Acids are titrated in the presence of indicators which change under slightly alkaline conditions.
- Bases should be titrated in the presence of indicators which change under slightly acidic conditions

The equivalence point of a titration

- When you carry out a simple acid-base titration, you use an indicator to tell you when you have the acid and alkali mixed in exactly the right proportions to "neutralize" each other. When the indicator changes color, this is often described as the *end point* of the titration.
- In an ideal world, the color change would happen when you mix the two solutions together in exactly equation proportions. That particular mixture is known as the *equivalence point*.

Example

- If you were titrating sodium hydroxide solution with hydrochloric acid, both with a concentration of 1 mol dm^{-3} , 25 cm^3 of sodium hydroxide solution would need exactly the same volume of the acid - because they react 1 : 1 according to the equation.



- In this particular instance, this would also be the *neutral point* of the titration, because sodium chloride solution has a pH of 7.

- This is not necessarily true of all the salts you might get formed.
- If you titrate ammonia solution with hydrochloric acid, you would get ammonium chloride formed. The ammonium ion is slightly acidic, and so pure ammonium chloride has a slightly acidic pH.
- That means that at the equivalence point (where you had mixed the solutions in the correct proportions according to the equation), the solution wouldn't actually be neutral. To use the term "neutral point" in this context would be misleading.

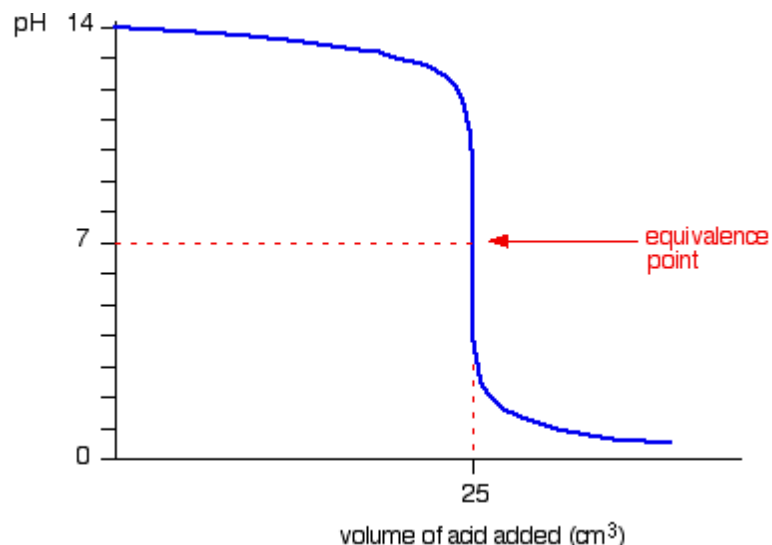
- Similarly:
- If you titrate sodium hydroxide solution with ethanoic acid, at the equivalence point the pure sodium ethanoate formed has a slightly alkaline pH because the ethanoate ion is slightly basic.
- $\text{NaOH} + \text{CH}_3\text{COOH} \rightarrow \text{CH}_3\text{COONa} + \text{H}_2\text{O}$

- The term "neutral point" is best avoided.
- The term "equivalence point" means that the solutions have been mixed in exactly the right proportions according to the equation.
- The term "**end point**" is where the indicator changes color, and this isn't necessarily exactly the same as the equivalence point.

Titration curves for strong acid v strong base

HCl and NaOH are typical strong acid and strong base.

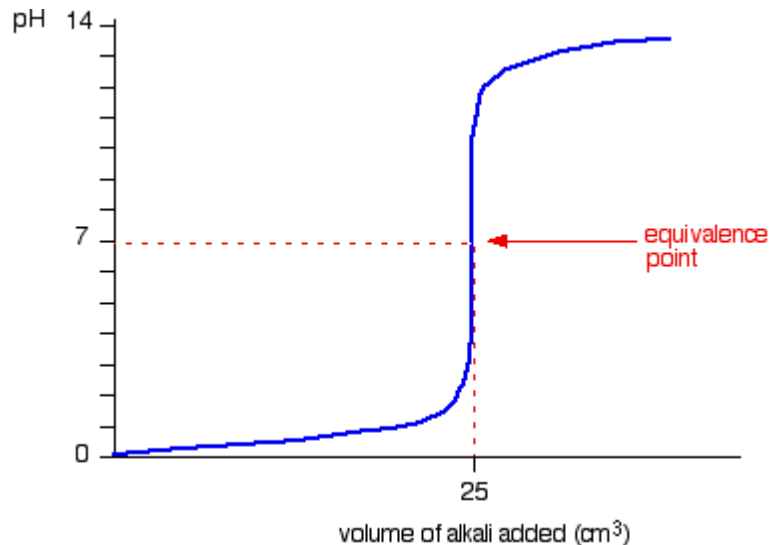
Running acid into the alkali



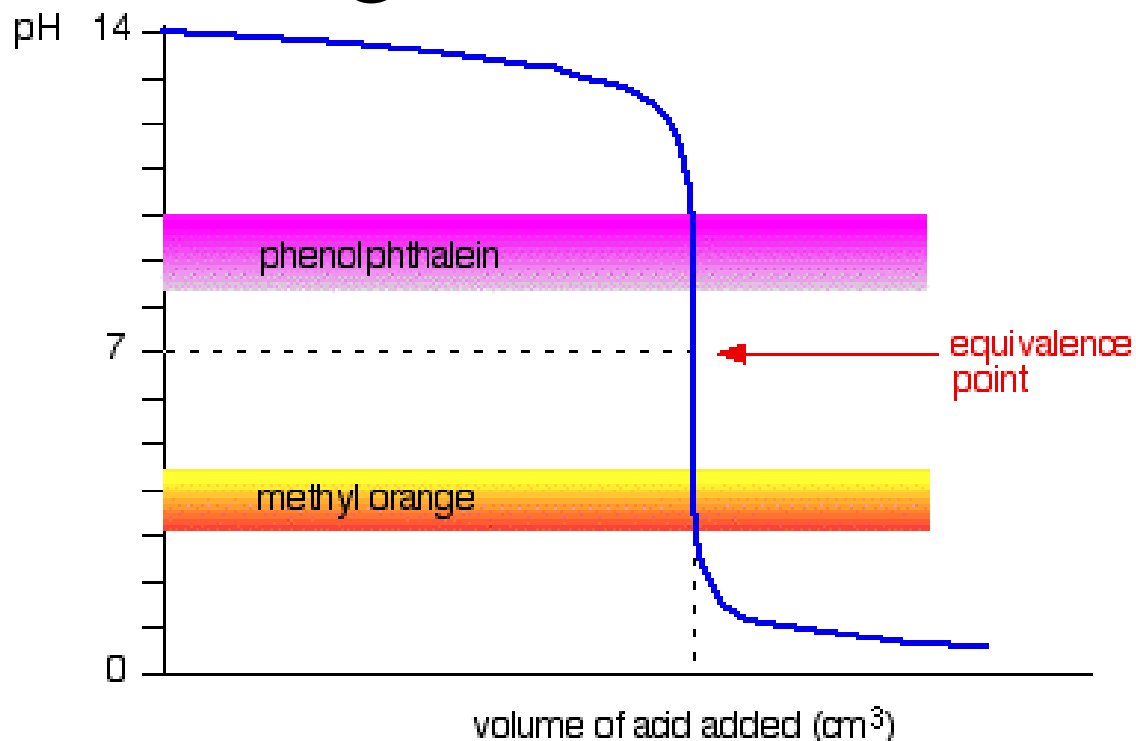
- You can see that the pH only falls a very small amount until quite near the equivalence point. Then there is a really steep plunge. If you calculate the values, the pH falls all the way from 11.3 when you have added 24.9 cm³ to 2.7 when you have added 25.1 cm³.

Alkali to Acid Titration

- This is very similar to the previous curve except, of course, that the pH starts off low and increases as you add more sodium hydroxide solution.



Titrating with an indicator



You can see that *neither* indicator changes color at the equivalence point. However, the graph is so steep at that point that there will be virtually no difference in the volume of acid added whichever indicator you choose. However, it would make sense to titrate to the best possible color with each indicator.

Strong Acids and Bases

- Don't confuse the words *strong* and *weak* with the terms *concentrated* and *dilute*.
- The strength of an acid is related to the proportion of it which has reacted with water to produce ions.
- The concentration tells you about how much of the original acid is dissolved in the solution.
- It is perfectly possible to have a concentrated solution of a weak acid, or a dilute solution of a strong acid.

Strong Acids and pH

- pH is a measure of the concentration of hydrogen ions in a solution.
- Strong acids like hydrochloric acid at the sort of concentrations you normally use in the lab have a pH around 0 to 1.
- The lower the pH, the higher the concentration of hydrogen ions in the solution

Standardization of NaOH

- A standard solution is one in which the concentration is known accurately.
- A standard solution of NaOH will be prepared.
- One way to prepare a standard solution is to dissolve an accurately massed amount of the substance and dilute it to a measured volume
- In this way, the concentration can be calculated exactly.
- $\text{Molarity} = \text{No of moles} / \text{Vol in liters}$

NaOH Standardization

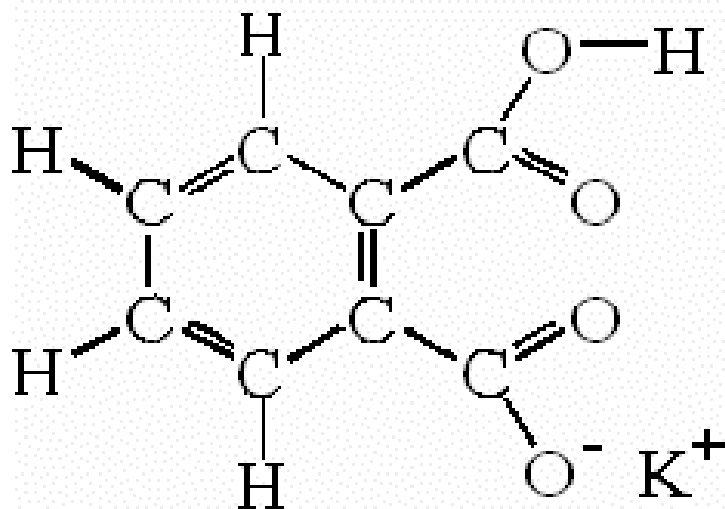
- It is usually impossible to obtain NaOH of sufficient purity to use it as a primary standard.
 - **Why?**
 - Sodium Hydroxide is hygroscopic (picks up water from the air)
- An indirect method is more practical for obtaining a standard solution of NaOH.
- A solution of a approximate molarity will be prepared and standardized against a **primary standard** of known purity.

What is a primary standard?

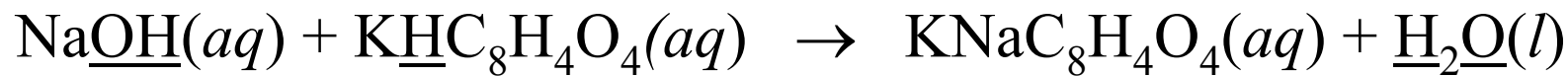
- A sample that is of high purity
- Remain unchanged in air during massing and remain stable during storage
- Have a high molar mass to reduce massing errors
- React with the solution to be standardized in a direct, well-defined reaction

- Potassium acid phthalate will serve as our primary standard.
- This is a large molecule ($\text{KHC}_8\text{H}_4\text{O}_4$) with a molar mass of 204.2 g/mol.
- Instead of writing the whole formula, it can be abbreviated as KHP, where "P" stands for the phthalate ion, $\text{C}_8\text{H}_4\text{O}_4^{2-}$, **not** for phosphorus.
- KHP is an acidic substance, with the ionizing hydrogen being set forward in the formula for emphasis.
- Therefore, KHP is monoprotic and will react with NaOH in a simple 1 to 1 relationship.

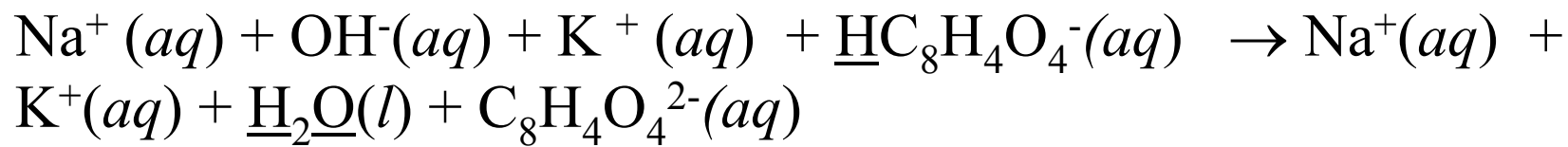
Potassium acid phthalate



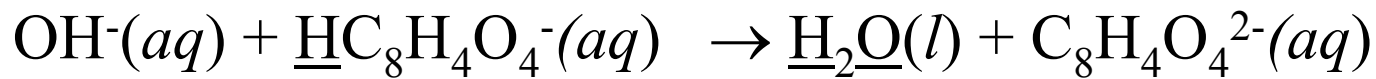
There is one acidic hydrogen (attached to the oxygen at top right corner) which can react with the base (OH⁻).



The ionic equation is:



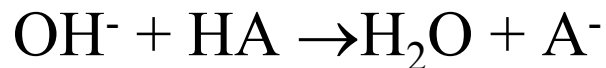
If the spectator ions (Na^+ , K^+) are removed:



This allows the following equation which applies at the endpoint* of the titration:

Moles acid used = Moles base used

Equivalence and End Points



- The equivalence point is when equal moles of OH^- have been added to HA because they react in a 1:1 ratio
- This can be difficult to determine, and instead use the end point
- Near the equivalence point of an acid base titration, the pH of the solution changes rapidly with relative amounts of HA and OH^- , and this changes the color of the indicator
- As the end point is approached a color change will be observed when 1 drop of titrant hits the analyte
- Try to reach the point where addition of 1 drop of titrant causes a permanent color change (> 10 secs) after swirling

Procedure

- The base (NaOH) will be standardized with KHP.
- This standard base will be used to standardize the acid so that the exact concentrations of both solutions will be known and can then easily titrate and analyze unknowns.

Procedure

- You will be given a solution of 0.1xx M NaOH.
- This will be your titrant.
- Rinse a buret with water and then with a small amount of the NaOH solution.
- Fill the buret with NaOH solution. Fill the buret tip by momentarily opening the stopcock. Read the initial volume.
- **Accurately** weigh approx. 0.8xx g of KHP into a 250 mL Erlenmeyer flask. Add about 100 mL of water and swirl the flask until the sample is dissolved.
- Add **3 drops** of phenolphthalein indicator (colorless in acidic solution; pink in basic solution).

- Titrate the KHP solution with the base solution to be standardized.
- Titration should proceed until the faintest pink persists for 30 sec. after swirling.
- The color will fade upon exposure to the air **(WHY?)**
- After completing a trial, breathe into the flask and swirl. What has happened?
- Make duplicate determinations and calculate the average molarity of the NaOH.
- For excellent work, the molarities need to be within 1% of one another.

Analysis of an Unknown Acid

- Obtain an unknown acid and perform 3 titrations
- You substitute your acid for KHP
- Check for precision of these titrations

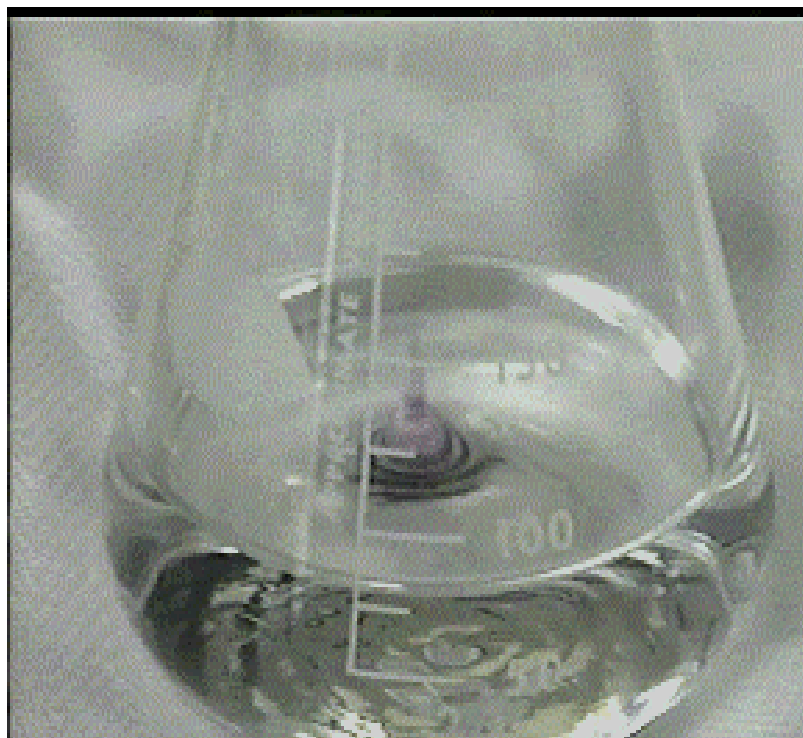
Data (for each trial):

- Mass of KHP in sample flask
- Initial buret reading
- Final buret reading
- Volume of NaOH required to neutralize the KHP

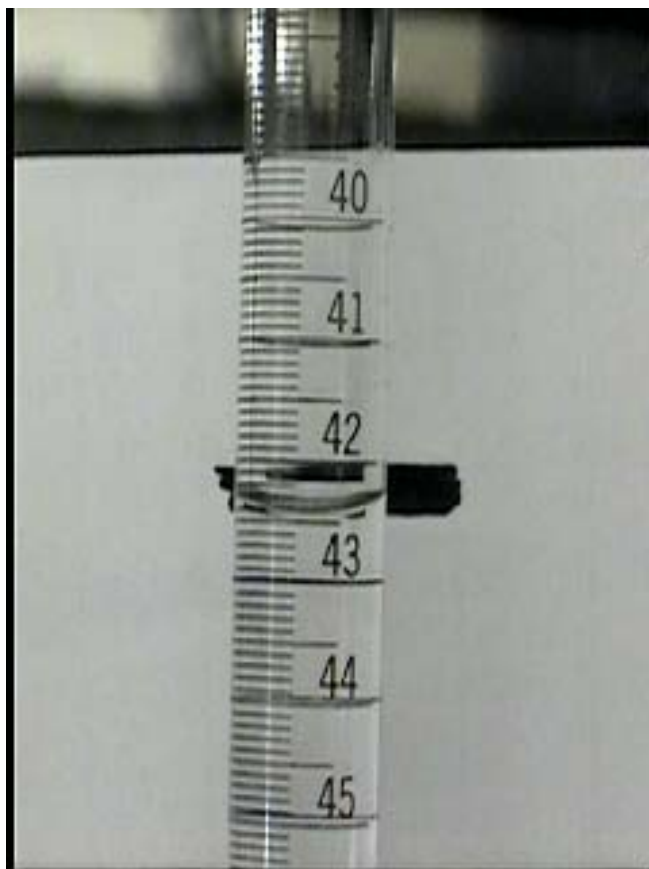
Experimental Techniques



Near the endpoint of the titration, you may want to add a fraction of a drop instead of a whole drop. To do this, let a little droplet grow out on the tip of the buret. Then wash it into the flask with a squirt of distilled water from your wash bottle



- You will know when you are getting close to the endpoint because addition of a small amount of NaOH (as little as a drop) will produce a pink color that will take a long time to fade as the flask is swirled.
- You have reached the endpoint of the titration when addition of one drop or less of solution causes the solution in the flask to turn pink and stay pink for 10 seconds or longer.



- Once the endpoint has been reached, read the final volume off of the buret. Here a volume of 42.5mls is read
- The volume of NaOH solution added to the flask is simply the final volume reading minus the initial volume reading.

Example Calculation

- Let's assume that we started with 0.700 g of KHP and it took 39.08 mL of NaOH to reach the endpoint, what value of [NaOH] do you get from this equation? I get a value of 0.08771 M.
- (Note: These numbers are totally bogus. When you do the experiment use your actual numbers for this calculation).

Example 2

- In a titration 23.25mls of NaOH , 0.125M was needed to react with 21.45 mls HCl.
- What is the molarity of the acid?

Example 3

- If 250mls of a 0.1M NaOH solution was required. How many grams would need to be weighed?

More Examples

- Calculate the molarity of a solution prepared by dissolving 4g NaOH in 100mls of solution?
- If 25mls of a 0.56M H₂SO₄ solution is diluted to a volume of 125mls. What is the molarity of the resulting solution?

Clean Up

- Dispose of excess NaOH solution in the 'surplus NaOH' bottle in the hood
- Dispose of excess acidic solutions in correctly labeled bottle
- Rinse the buret thoroughly with deionized water and place upside down in buret clamp
- Wipe down benches
- Put away equipment