

FE 211 ANALYTICAL CHEMISTRY

Experiment No. 2

NEUTRALIZATION TITRATIONS IN AQUEOUS SOLUTIONS

The neutralization of hydronium or hydroxide ion to form water is widely used as the basis for volumetric determinations of acids, bases and salt of weak acids. The reaction is characterized by a rapid change in pH near the equivalence point; a change that is readily detected by an acid-base indicator or that can be followed electrically by use of a pH meter.

Neutralization titrations are performed with standard solutions of strong acids or bases. In principle, a single solution (acid or base) is enough but in practice it is convenient to have both a standard acid and standard base available to locate end points more exactly. One solution is standardized against a primary standard. The molarity of the other is then found by determining the acid-base ratio (that is, the volume of acid required to neutralize 1.000 mL of base).

Atmospheric CO₂ is in equilibrium with aqueous carbonic acid, H₂CO₃, whose concentration is about 1.5x10⁻⁵ M at ordinary temperature. So this small concentration of carbonic acid leads to no significant error. On the other hand, the distilled water is sometimes supersaturated with the gas and thus contains sufficient carbonic acid to cause detectable errors. To test the water to be used in neutralization titrations; draw about 500 mL from the source, add 5 drops of phenolphthalein and titrate with 0.1 M NaOH. Less than 0.2 to 0.3 mL of base should be needed to form the first faint pink color. If a large volume is required, standard solutions should be prepared from water that has been boiled briefly to remove CO₂ and cooled to room temperature.

PREPARATION AND STANDARDIZATION OF APPROXIMATELY

0.1 M SOLUTIONS OF HCl AND OF NaOH

Experimental:

A) Reagents:

1. Hydrochloric acid, HCl, approximately 0.1 M solution prepared by diluting 8.3 mL stock solution (specific gravity 1.19 and 37 % HCl) to 1 liter with water. (or Prepare a hydrochloric acid solution from given solution).
2. Methyl red indicator.
3. Phenolphthalein indicator

4. Sodium hydroxide, NaOH, approximately 0.1 M solution, prepared by dissolving 4 grams NaOH in water and diluted to 1 liter. (or Prepare a sodium hydroxide solution from given solution).
5. Potassium hydrogen phthalate, $\text{KHC}_8\text{H}_4\text{O}_4$, (KHP), reagent grade.
6. Tris- (hydroxymethyl) aminomethane, $\text{C}_4\text{H}_{11}\text{NO}_3$.
7. Methyl orange indicator.
8. Vinegar.

Standardization of 0.1 M NaOH

1. Dry a quantity of primary-standard potassium hydrogen phthalate, for 3 hr at 110°C and cool in a desiccator.
2. Weigh 0.7 to 0.9 g samples into 250 mL conical flask, and dissolve in 75 mL of freshly boiled and cooled distilled water.
3. **or Take exactly 10.0 mL prepared KHP solutions into an erlenmeyer flasks and dilute to 100 mL with water.**
4. Add 2 drops of phenolphthalein and titrate with base until pink color of the indicator persists for 30 sec.
5. Calculate the molarity of NaOH solution.

Standardization of 0.1 M HCl :

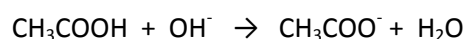
1. Dry a quantity of primary standard tris- (hydroxymethyl) aminomethane for 2 hours at 110°C and cool in a desiccator.
2. Weigh two portions 0.5 to 0.7 gram samples into 250 mL erlenmeyer flasks.
3. Add 100 mL water and 2 drops methyl red indicator solution.
4. Shake by hand until the salt has dissolved
5. **or Take exactly 10.0 mL of tris- (hydroxymethyl) aminomethane solutions into erlenmeyer flasks and dilute to 100 mL with water.**
6. Add 2 drops of methyl orange indicator.
7. Titrate with HCl until the solution just turns red.
8. Calculate the molarity of the acid solution. Replicate results should agree within 2 or 3 parts per thousand.

Determination of Acetic Acid in Vinegar :

1. Weigh a glass-stoppered weighing bottle. Insert about 5 mL of the vinegar sample and reweigh the bottle. **(or Take 5.0 mL from prepared solution).**

2. Transfer the sample completely to a 250 mL erlenmeyer flask and dilute with 100 mL of previously boiled distilled water.
3. Add three drops of phenolphthalein solution.
4. Run in standard alkali, NaOH, from a buret until a pink color just persists. If too much base is added, add standard acid dropwise from a second buret until the color disappears. Then add more base from the first buret until the indicator color just returns again.
5. Repeat the titration with two separately weighed portions of the vinegar or prepared solution.
6. Calculate the weight percentage acetic acid, HC₂H₃O₂, in the sample.

The net reaction during titration is;



Analysis of the Mixture of Strong and Weak Acids

Analysis of two monoprotic acids can be done by titration with a strong base. If one of the acids is strong, other weak and K_a for weak acid is 10^{-4} or less, there are two end points in the titration and titration can be done by using two different indicators. An example of this type of analysis is the analysis of the mixture of HCl and acetic acid, HC₂H₃O₂. In the process first HCl and then HC₂H₃O₂ is titrated.

Procedure:

1. Take exactly 10.0 mL of your unknown sample and dilute to about 100 mL with water.
2. Add 2 or 3 drops methyl orange indicator and titrate with standard NaOH solution until the color changes, and then add 2 drops of phenolphthalein indicator and titrate till the appearance of pink color.
3. Determine the amount of HCl from the volume and molarity of NaOH used to obtain first end point (methyl orange end point).
4. Determine the amount of HC₂H₃O₂ from the volume and molarity of NaOH used to get second end point with phenolphthalein.

FE 211 ANALYTICAL CHEMISTRY DATA SHEET

Experiment No: 2

NEUTRALIZATION TITRATION IN AQUEOUS SOLUTIONS

1) Standardization of 0.1 M NaOH

Volume of NaOH used (mL) :

2) Standardization of 0.1 M HCl

Volume of HCl used (mL) :

3) Determination of Acetic Acid in Vinegar

Volume of NaOH used (mL) :

4) Analysis of Mixture of Strong and Weak Acids

Volume of NaOH used to obtain first end point (mL) :

Volume of NaOH used to obtain second end point (mL) :

Submitted by:

Submitted to:
Date: