

pH-metric titration: determination of the strength of a given hydrochloric acid solution against a standard sodium hydroxide solution

Introduction

Most of the chemical and biochemical processes are profoundly affected by the acidity or alkalinity of the medium in which the reaction takes place. All acids dissociate in aqueous solution to yield H^+ ions. Some acids like HCl , H_2SO_4 , HNO_3 etc. are completely ionized in aqueous medium whereas CH_3COOH , $HCOOH$ etc. ionize to a small extent only. The former is known as strong and the latter as weak acid. pH of any solution is defined as $(-\log H^+)$ and has values between 0–14. $pH < 7$ indicate acidic solution, $pH > 7$ indicate basic solution and $pH = 7$ means neutral solution.

The pH meter is an instrument that measures the pH of a solution and affords a direct method of obtaining a titration curve. A titration curve is a graph of measured pH values versus the volume (milliliters) of titrant added. **Figure 1** below is an example of a titration curve, illustrating the numerous data points and the best smooth curve drawn through the points.

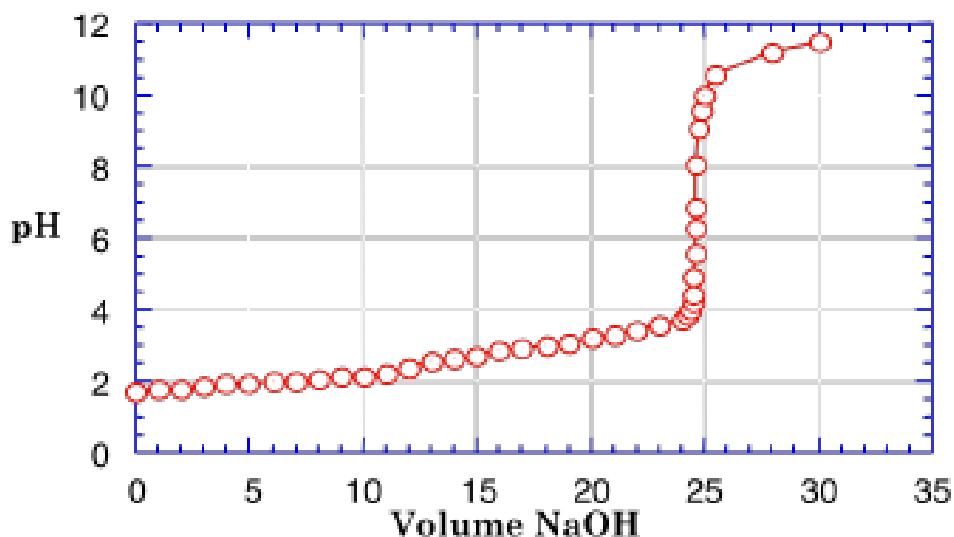


Figure 1. Titration Curve of acid HA

The equivalence point is the point at which an equal amount of acid has been added to the amount of base present or vice versa. The equivalence point occurs on the titration curve in the region where there is a relatively large change in pH with a relatively small change in volume. The steeper the curve in the region of the equivalence point the more precisely it may be established. Once a titration curve is constructed and the equivalence point established the experimenter could then choose an indicator that would give a suitable endpoint (point at which the indicator changes color).

Selection of the equivalence point

In this experiment you will graph the measured pH against the volume of standard (~ 0.10 M) NaOH solution added. The best smooth curve should be drawn through these points. The equivalence point can be established using the steepest tangent to the smooth curve where the pH changes rapidly. The equivalence point is the point where the line intersecting the volume axis. The method is summarized in **Figure 2** below. The equivalence point selected using this method is a more accurate method than using an indicator in the titration.

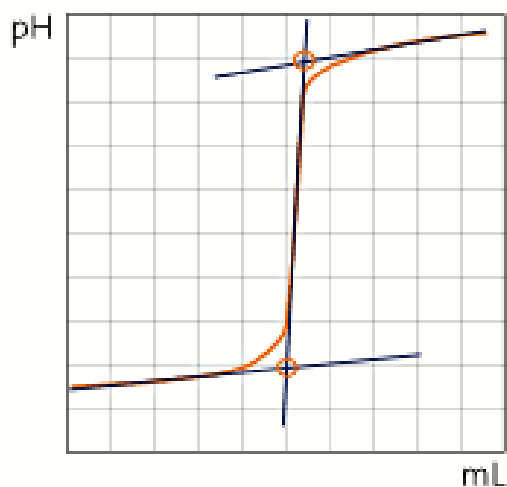


Figure 2. Equivalence point determination for acid HA

A second method maybe used to determine the equivalence point. To use this method a graph is constructed of $\Delta\text{pH}/\Delta V$ vs. V average . **Figure 3** below illustrates this method.

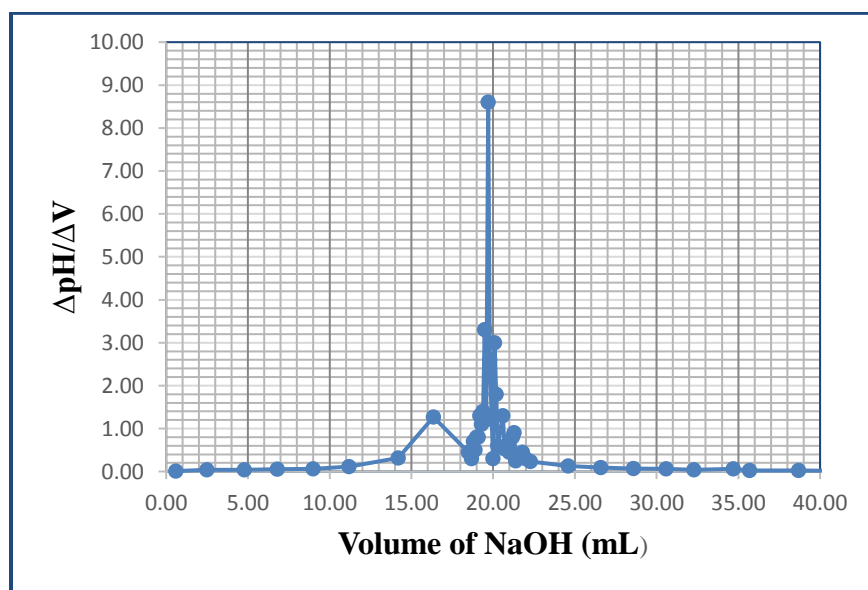


Figure 3. Equivalence point determination for acid HA using the first derivative method.

The volume at the point where the graph reaches the maxima is the equivalence point of the titration. A disadvantage of the titration curve method is the time and effort required to make the measurements and to construct the graph. This disadvantage can be overcome by using a recording pH meter, which provides a chart record of the pH of a solution as a function of time.

Procedure

A. Rough titration: run a rough titration at first to obtain an estimate of the volume of NaOH needed to reach the equivalence point.

1. Use volumetric pipet to transfer 25.00 ml of HCl to a 250-ml flask (use HCl previously prepared in Exp. #2, we will consider it as an unknown), add 3-5 drops of phenolphthalein indicator to the same flask.
2. Fill the buret carefully to 0.00 ml with NaOH previously prepared in Exp. #1. Place the flask from step 1 beneath the buret and start the titration process to the first faint pink end point, record down the volume of NaOH needed to reach this point.

B. pH metric titration

1. Calibrate the pH-meter by immersing the electrode in a reference buffer solution.
2. Use volumetric pipet to transfer 25.00 ml of HCl to a beaker (use HCl previously prepared in Exp. #2, we will consider it as an unknown), then immerse the pH-meter electrode in the HCl solution.
3. Titrate potentiometrically with the standard NaOH solution. Add rather large increments (around 2ml) of base at first. Add smaller increments as the end point is approached. After each increment, stir well and record the volume of titrant and the pH at this point.
4. If time permits, repeat steps 2 and 3 above two more times to obtain a total of three replicate measurements.

Report

1. For each replicate, construct a titration curve (Full titration curve) on a graph paper with pH on the vertical axis and milliliters 0.10M NaOH added on the horizontal axis,
2. Following the instructions described in Figure 2, construct the expanded titration curve to determine the equivalence point (do this for one replicate at least),
3. To determine the equivalence point accurately, construct the first derivative curve by plotting $\Delta\text{pH}/\Delta V$ vs. V-NaOH added (do this for three replicates if possible).

- Using the volume of standard NaOH solution at the equivalence point, and its average molarity (from Expt. # 1), calculate the molar concentration of HCl solution ($\text{HA}_{(\text{aq})} + \text{NaOH}_{(\text{aq})} \rightarrow \text{NaA}_{(\text{aq})} + \text{H}_2\text{O}_{(\text{l})}$).

Statistical Analysis of Data

- Calculate the molar concentration of HCl for the three replicate measurements,
- Calculate average molar concentration of HCl,
- Calculate standard deviation associated with the average molar concentration of HCl,
- Conduct Q_{test} or G_{test} to check for an outlier in the three calculated molar concentrations of HCl,
- Calculate confidence interval at 95% confidence limit.

Calculations:

	Trial 1	Trial 2	Trial 3
mL of NaOH at equivalence point	_____	_____	_____
Concentration of HCl Solution (M)	_____	_____	_____

G -test

Average concentration of HCl Solution

Standard deviation (s)

Confidence Interval at 95% confidence level

Questions:

1. What are some sources of errors (random or systematic) in this experiment?
2. From your experience, what are the shortcomings of this experiment that could be improved next semester?
3. Which method is more accurate in determining the equivalence point, the first derivative method? or drawing a tangent line at the expanded calibration curve?