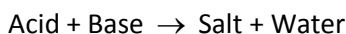


Experiment 7 - Acid-Base Titrations

Titration is an analytical method used to determine the exact amount of a substance by reacting that substance with a known amount of another substance. The completed reaction of a titration is usually indicated by a color change or an electrical measurement. An acid/base neutralization reaction will yield salt and water. In an acid-base titration, the neutralization reaction between the acid and base can be measured with either a color indicator or a pH meter.



In this experiment, a phenolphthalein color indicator will be used. Phenolphthalein is colorless in acidic solutions and pink in basic solutions. Phenolphthalein is also used in forensic crime scene analysis to detect the presence of blood, Kastle-Meyer test. In the Kastle-Meyer test, hemoglobin catalyzes the oxidation of the colorless form of phenolphthalein to its bright pink form.

Four lab periods assigned for this experiment. In part I you will prepare an acid (HCl) solution and a base (NaOH) solution. These solutions will be used for all four periods so it is important to keep these solutions. These solutions will be titrated against each other to obtain a base/acid ratio. In part II you will find the normality of the base solution by titrating it against a solid acid standard. The normality of the acid can be calculated from the normality of the base and the base/acid ratio from part I. In part III the base will be titrated against an unknown acid to find the equivalent weight of the acid. In part IV the equivalent weight of an unknown base will be determined by reacting the unknown base with an excess of HCl and "back-titrating" the left-over acid with NaOH.

Equipment and Reagents (Part I)

6 NHCl	1 Liter plastic bottle	2 beakers (50 mL)
6N NaOH	2 burets	250 mL Erlenmeyer Flask
500 mL Florence Flask	Iron stand	wash bottle
Distilled water	buret clamp	phenolphthalein indicator
Stopper(or parafilm)	2 x 50 mL graduated cylinder	

Procedure (Part I)

1. Rinse a clean 500 mL Florence flask with a small portion of DI water. Place about 16-17 mL of 6 M or 6 N HCl into the flask and dilute to 500 mL with distilled water. The 500 mL is approximated by bringing the level of the solution up to the point of constriction of the neck of the flask. Stopper the flask and shake to mix. The solution should be approximately 0.2 N HCl. Label the flask with tape.
2. Rinse a clean 1 L plastic bottle with distilled water. Place about 32-34 mL of 6 M or 6 N NaOH into the bottle and dilute to 1 liter with distilled water. Place the cap on the bottle and shake to mix. The solution should be approximately 0.2 N NaOH. Label the bottle with tape.

- Obtain 2 burets from the stockroom and clamp them onto the ring stand using the buret clamp. Label the buret as acid or base. Label the 50 mL beakers as acid or base. These beakers will be used to transfer the solutions into the burets. Rinse each buret with about 5 mL of DI water and with about 3 x 5 mL of the solution to be used. Fill each buret with the correct solution and flush all of the air bubbles out of the buret tip.
- Read the initial level of each buret to the nearest 0.02 mL and record this in your notebook. The proper reading is taken from the bottom of the meniscus (see Figure 1 below). If the initial reading is at exactly at zero, then report 0.00 mL.

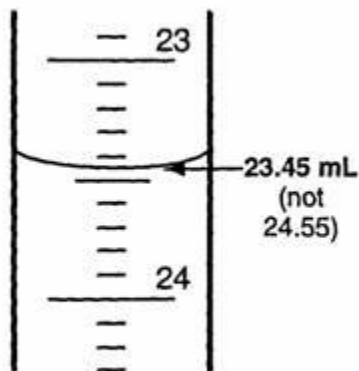


Figure 1. How to read a buret volume.

- Allow about 25 mL of the acid to run into an Erlenmeyer flask from the acid buret. Record the initial and final readings of this transfer. Calculate the volume of acid transferred by subtracting the final volume reading by the initial volume reading. Your final answer should be to the hundredth place.
- Add about 20 mL of distilled water into the flask and add 2-3 drops of phenolphthalein indicator. The flask should remain colorless at this point.
- Record the initial volume of base. Slowly add NaOH from the base buret into the flask with constant swirling. Continue adding base until a very faint color remains. If the color is too bright, add a few drops of acid so that the solution becomes colorless. Add base again to reach the faint end-point. Repeat this process until a faint pink end-point is reached. Record the final volume of base and the initial and final volume of extra acid added to this flask
- Calculate the total final volume of acid and final volume of base added.
- From these values, calculate the base to acid ratio:

$$\text{base to acid ratio} = \frac{\text{volume of base}}{\text{volume of acid}}$$
- Re-fill the burets and repeat the procedure 2 more times for a total of 3 trials.
- Calculate the average value for the base/acid ratio.

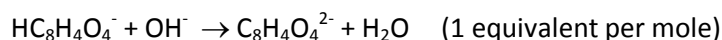
Equipment and Reagents (part II)

Your HCl solution	2 burets	250 mL Erlenmeyer flask
Your NaOH solution	Iron stand	2 beakers (50 mL)

Potassium phthalate	phenolphthalein	100 mL graduated cylinder
Weighing paper	buret clamp	

Procedure (Part II)

1. Clean a 250 mL Erlenmeyer flask and then rinse with DI water.
2. On the analytical balance, weigh between 1.0-1.4 grams of potassium phthalate (KPh) or $\text{KHC}_8\text{H}_4\text{O}_4$ (203 g/mol) to the nearest 0.0001 g on a piece of weighing paper. Record the exact mass.
3. Transfer the KPh to the 250 mL flask and add about 50 mL of DI water and swirl to dissolve. The solids must be completely dissolved. Add 2-3 drops of phenolphthalein indicator.
4. Re-fill the burets with the appropriate solution. Record the initial volume readings and titrate the sample to the faint pink end-point. Record the final volume readings.
5. Repeat the procedure for a total of 2 trials.
6. Calculate the normality of the NaOH solution for each trial and average these results.



$$N_b = \frac{\text{equivalents NaOH}}{\text{volume of NaOH}}$$

Make sure to use the corrected value for NaOH if there was a HCl addition using the base/acid ratio from part 1:

$$\text{Vol}_{\text{base (extra)}} = \text{Vol}_{\text{acid}} \times \frac{\text{volume of base}}{\text{volume of acid}}$$

$$\text{Vol}_{\text{base(total)}} - \text{Vol}_{\text{base(extra)}} = \text{Vol}_{\text{corrected}}$$

7. Calculate the normality of the HCl solution using the base/acid ratio from part 1.

$$N_a = N_b \times \frac{\text{volume of base}}{\text{volume of acid}}$$

Equipment and Reagents (Part III)

Unknown solid acid	2 burets	2 beakers (50 mL)
Your solutions	250 mL flask	buret clamp
Phenolphthalein indicator	Iron stand	weighing paper

Procedure (Part III)

1. Obtain an unknown solid acid and record the ID number.
2. Weigh between 0.8-1.0 grams of the unknown on the analytical balance.

3. In a 250 mL flask. Dissolve each sample in about 50 mL of distilled water and add 2-3 drops of indicator.
4. Titrate the sample as before.
5. Repeat the procedure for a total of 2 trials.
6. Calculate the equivalent mass for each trial and average the results.

$$\text{equivalents of acid} = \text{equivalence of base} = V_{\text{base}} \times N_{\text{b}}$$

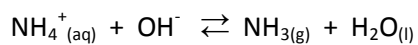
$$\text{Equivalent mass} = \frac{\text{mass of sample}}{\text{equivalents of acid}}$$

Equipment and Reagents (Part IV)

Unknown ammonium salt	buret clamp	bromothymol blue indicator
250 mL Erlenmeyer Flask	2 beakers 50 mL	funnel
2 Burets	Your Solutions	red litmus paper
Iron Stand	hot plate	

Procedure (Part IV)

1. Obtain a sample of unknown ammonium salt. Record the unknown ID in your notebook. On the analytical balance weigh about 0.3 g of the unknown salt into a 250 mL Erlenmeyer flask.
2. From your base buret add about 50 mL of the standard NaOH to the sample. Record the initial and final buret readings. Calculate the volume of base added to the sample.
3. Place a funnel in the neck of the Erlenmeyer flask to prevent any loss of the solution. Dissolve the salt and boil the solution gently on the hot plate. *Keep the splattering of the solution to a minimum when boiling.*
4. After about 5 – 10 minutes check for the presence of NH₃ in the steam coming from the mouth of the flask. To do this, hold a piece of moist red litmus paper over the stream of vapor leaving the flask. A blue color indicated that NH₃ is still present in the solution. Continue boiling until a negative test result is obtained (red litmus should remain red).
5. When no more ammonia is present, remove the flask from the hot plate and rinse any condensed liquid from the funnel with DI water back into the flask. Add DI water to make a volume of about 50 mL.
6. Add about 10 drops of bromothymol blue indicator to each solution and titrate the excess NaOH with the standard HCl solution. The initial color should be blue and the end-point should be yellow. A faint green-yellow color is the ideal end-point. Record the initial and final buret readings from the HCl addition. Calculate the volume of HCl transferred.
7. Repeat the procedure for a total of 2 trials.
8. Calculate the equivalent mass of the base.



Total equivalents of base = $V_b \times N_b$

Equivalents of acid = $V_a \times N_a$

Equivalents of base used up = Total equivalents – equivalents of acid

At the end-point = equivalents of base = equivalents of NH_4^+

$$\text{Equivalent Mass} = \frac{\text{mass of sample}}{\text{equivalents of } \text{NH}_4^+}$$

Report

Report the average normality for the standardized solutions.

Report the average equivalent mass for the unknown solid sample and included the unknown ID.

Report the average equivalent mass for the unknown ammonium base sample and include the unknown ID.