Standardization by Titration	Identifier: CSP-0018 Revision: 0 Page: 1 of 5

ISU Chemistry Department	Stockroom Procedure	Effective Date: 5/1/2023

1. INTRODUCTION

This procedure guides the standardization of an aqueous base solution against a primary standard acid, and the standardization of an aqueous acid against a standardized aqueous base.

2. PRECAUTIONS AND LIMITATIONS

2.1. Add acid or base to water.
2.2. Wear proper protective equipment.

3. APPARATUS AND MATERIALS

3.1. 50 mL class A burette
3.2. Erlenmeyer flasks, various sizes
3.3. Beakers, 100 & 250 mL
3.4. Analytical balance
3.5. Magnetic stir bar
3.6. Magnetic stir plate
3.7. Short stem funnel
3.8. Oven set to 120^{o}C
3.9. Class A volumetric pipettes, various sizes dependent on solution concentrations
3.10. Gloves
3.11. Scoopula
3.12. Pipette filler (bulb or roller)

4. REAGENTS

4.1. Primary standard Potassium Hydrogen Phthalate, KHP.
4.2. Sodium hydroxide pellets
4.3. Phenolphthalein indication solution, 1%
4.4. Deionized water (DIW)

5. INSTRUCTIONS

5.1. Preparing the primary standard KHP:
5.1.1. Place an excess amount of the primary standard KHP powder into an oven set to 120^{o}C in an uncovered 100mL beaker
5.1.2. Let dry for 2 to 3 hours.

5.1.3. Cool dried primary standard KHP in a desiccator for at least 1 hour, but not more than 24 hours.

5.1.4. Using an analytical balance measure 4 aliquots of the primary standard KHP into 4 125 mL Erlenmeyer flasks labeled for identification.

5.1.4.1. Record the weight of KHP in each flask in the laboratory notebook.

Note 1: *Three aliquots are necessary for calculations. Measuring 4 aliquots provides an extra sample in the event one aliquot is discarded for any reason.*

Note 2: *The mass of KHP used will depend on the concentration of the basic solution being standardized.*

Example 1: *For a 0.1 M solution of sodium hydroxide, measure between 0.5-0.6 grams of KHP to keep the volume of solution needed to titrate the solution between 25-35mL.*

Example 2: *For a 0.8 M solution of sodium hydroxide, measure between 1.8-2.1 grams of KHP to keep the volume of solution needed to titrate the solution between 25-35mL.*

Note 3: *Remove the Erlenmeyer flask from the balance chamber to scoop the KHP into the flask. Return the flask to the balance chamber, close door and record the weight 10 seconds after the stable symbol appears on the balance screen. Consistency in every step is key to successful KHP titrations.*

5.1.5. Fill the Erlenmeyer flasks with deionized water (DIW) to the 50 mL mark on the flask

5.1.6. Add a 1" magnetic stir bar to each flask

5.1.6.1. Stir each flask on a magnetic stir plate until solid dissolves, rinsing down the sides of the flask with a squeeze bottle of DIW to ensure all KHP crystals are in solution.

5.1.6.2. Stir the solutions for approximately 10 minutes to fully dissolve the KHP.

5.1.6.3. Rinse down sides of the flask a final time to rinse any of the solution that may have splashed up on the flask sides into the final solution.

5.1.7. Add 3-5 drops of 1% phenolphthalein to each flask.

5.2. Titrating the primary standard KHP with the base solution:

5.2.1. Fill a 50 mL class A burette with sodium hydroxide solution allowing ample time for solution to completely run down the sides of the burette before taking initial volume reading to 2 places past the decimal.

5.2.2. Titrate KHP with sodium hydroxide solution while constantly stirring on a stir plate until a faint pink color persists for 30 seconds.

Note 1: *Run the NaOH solution from the burette in a stream until observing a distinct pink persisting at the vortex of the KHP solution in the flask that disappears quickly. Slow down the titration to a rapid drop as the pink lengthens its persistence time, then a slow drop, then a dropwise addition.*

Note 2: *If the solution is magenta, it is over titrated. You must discard it and start again. Hence, 4 aliquots are measured for a safety net.*

5.2.3. Record final volume reading from burette to 2 places past the decimal.

5.2.4. Repeat steps 5.2.1-5.2.3 for all KHP solutions.

5.3. Titrating an acid of approximate concentration with a standardized base solution:

5.3.1. Pipette 4 aliquots of acid into separate, labeled Erlenmeyer flasks.

Note*: Size of the pipette depends on the concentration of the standardized base and the approximate concentration of the acid. Choose a pipette volume that will use at least 10 mL of standardized base, but not more than 45mL.*

Example 1*: If the standardized base is 0.1105M and the acid concentration is approximately 0.1M, choose a 15 or 25mL pipette. The titration will take about an equal amount of base as acid pipetted to reach the equivalence point.*

Example 2: *If the standardized base is 0.1105M and the acid concentration is approximately 0.2M, use a 10 or 15mL pipette. It will take about 2X the volume of base as the amount of acid pipetted to reach the equivalence point of the acid because the acid concentration is 2X that of the base.*

***Example 3**: If the standardized base is 0.1105M and the acid concentration is approximately 0.01M, pipette 100 mL of the acid. The base is 10X more concentration than the acid, so 10X less base will be needed to reach the acid equivalence point. In this case, only about 10 mL of the base will be needed to reach the equivalence point of 100mL of the acid.*

5.3.2. Add 3-5 drops of indicator to each flask.

Note 1: *Choose an indicator that changes color in the pH range of the equivalence point. Charts are available online or on the door of the indicator cabinet in the stockroom.*

Note 2: *The two indicators used by the stockroom for acid concentration determination are phenolphthalein and Bromothymol blue (used for Chloroacetic acid).*

5.3.3. Fill a 50 mL class A burette with sodium hydroxide solution allowing ample time for solution to completely run down the sides of the burette before taking initial volume reading of the burette to 2 places past the decimal.

5.3.4. Titrate the acid with sodium hydroxide solution while constantly stirring on a stir plate until a faint pink color persists for 30 seconds.

5.3.5. Record final volume reading from burette to 2 places past the decimal.

5.3.6. Repeat steps 5.3.3 to 5.3.5 for all acid samples.

5.3.7. Follow equations in section 7 to calculate the acid and/or base concentrations needed.

5.4. CLEANUP

5.4.2. Drain burette contents into a beaker.

5.4.3. Fill burette with DIW and drain completely.

5.4.4. Repeat rinsing burette with DIW 2 more times for a total of 3 rinsings

5.4.5. Neutralize all burette rinsings with 5% sodium bicarbonate solution and discard in drain.

5.4.6. Pour all titrated acids and bases down drain.

5.4.7. Clean all glassware following the Glassware Washing procedure CSP-0005.

6. CALCULATIONS

6.1. Base concentration in molarity from standardization against primary standard KHP (M_B)

$moles_b = G_k * (1/F_w)$
$M_b = moles_b/L_b$

Where:
M_b = concentration of standardize base in molarity
$moles_b$ = total moles of base being titrated
G_k = weight of KHP used in grams
F_w = formula weight of KHP = 204.22 g/mol
L_b = volume of base used in titration in liters

6.2. Acid concentration in molarity from titration against standardized base (M_A)

$moles_a = M_b * L_b$
$M_a = moles_a / L_a$

Where:
M_a = concentration of standardized acid in molarity
$moles_a$ = total moles of acid being titrated
M_b = molarity of standardize base from equation 7.1
L_b = volume of based used in titration in equation 7.1 in liters
L_a = volume of acid used in titration in liters

NOTE: *Equations 6.1 and 6.2 assume a balanced 1 to 1 ratio of moles in the calculation (i.e. 1 mol base/1mol KHP in 6.1) and should be adjusted accordingly in situations that are not 1 to 1.*