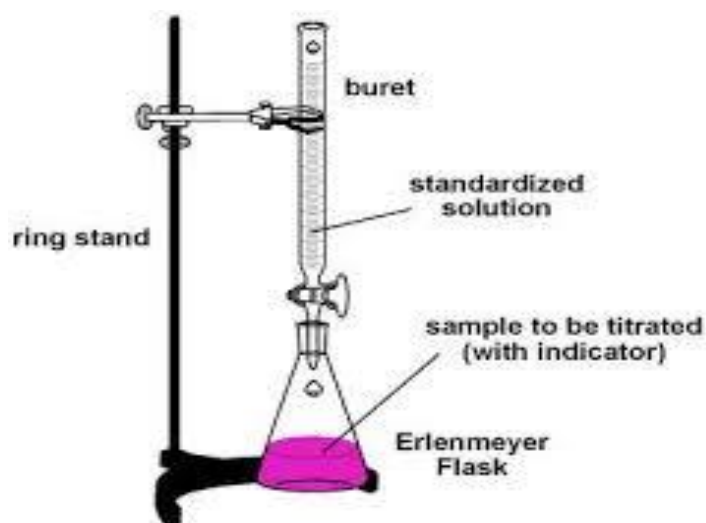


FACULTY OF
HUMANATIES & SCIENCE

Module Name	Laboratory safety and management
Module Code	SC1162
Title of Experiment	Preparation of a primary standard solution and titration of a secondary standard solution using it.
Number of the Experiment	03
Name of the Student	Liyanage L.H.K.
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Objectives:- To introduce primary standards and secondary standards.

To introduce the proper technique of preparing a standard solution.

To use primary standard solutions to determine the concentration of secondary standard solutions in titrimetric.

Introduction:-

Titration is an analytical method used to determine the exact amount of a substance by reacting that substance with a known amount of unknown amount. The completed reaction of a titration is usually indicated by a color change of end point.

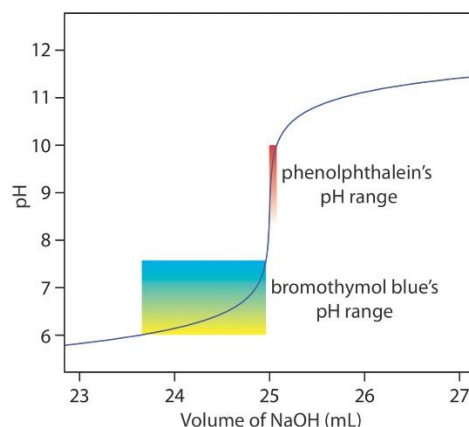
Primary standard solution is made out of primary standard substance. A primary standard is a substance of known high purity (99.9% about pure) which may be dissolved in a known volume of solvent to give a primary standard solution. Primary standard is reagent that can involve in chemical reaction. Primary standard should be high purity, high stability, high equivalent weight, not hygroscopic, inexpensive, low toxicity, readily available. Sodium carbonate (Na_2CO_3), Potassium hydrogen iodate ($\text{KH}(\text{IO}_3)_2$), Potassium dichromate ($\text{K}_2\text{Cr}_2\text{O}_7$), Potassium hydrogen phthalate or KHP ($\text{C}_8\text{H}_5\text{KO}_4$) some of examples of primary standard solution.

A secondary standard solution is a solution that is made specifically for a certain analysis. It is usually standardized against a primary standard. Usually there are some properties of secondary standard solution and they are not very pure, reactive than primary standards, they are somewhat hygroscopic. Potassium permanganate (KMnO_4), Sodium hydroxide (NaOH), Potassium hydroxide (KOH), Hydrogen sulphate (H_2SO_4) are some of examples of secondary standard.

A commonly used primary standard for titration with sodium hydroxide solution (NaOH). Potassium hydrogen phthalate or KHP is the weak acid NaOH is strong base. In this reaction as well, one molar of KHP completely reacts with one mole of NaOH . The titration of NaOH with KHP involves adding KHP from the burette to a unknown concentration of NaOH .



A suitable indicator for the titration of the weak acid KHP and the strong base NaOH would be either bromothymol blue (BTB) range (6.0-7.6) or phenolphthalein (8.3-10.0). BTB reagent is yellow in acidic solution, blue in basic solution and green in neutral solution. pH reagent is colorless in acidic solution, pink in basic solution. In here Bromothymol blue and Litmus cannot use as a indicator, because its' pH range of color change less. So then the end point could not see.



Material and methods:-

Material:-

potassium hydrogen phthalate solution, sodium hydroxide solution, phenolphthalein indicator, burette, pipette, titration flask, funnel, distilled water, glass rod, dropper

Procedure:-

Preparation of a 1.0×10^{-2} mol dm^{-3} potassium hydrogen phthalate (KHP) solution.

0.51g mass of Potassium hydrogen phthalate (KHP) was calculated by analytical balance. A beaker was added KHP solid and was added distilled water it. Then the solid was dissolved. Then 250cm^3 volumetric flask was transferred it. The solid was dissolved completely by using distilled water. After distilled water was added up to the mark and inverted the volumetric flask several times.

Determining the concentration of NaOH solution using previously prepared potassium hydrogen phthalate solution.

Firstly the burette was rinsed with tap water and with distilled water. Next the burette was rinsed with KHP solution. The burette was filled with 1.0×10^{-2} mol dm^{-3} KHP solution and the initial burette reading was recorded. A volume of 25.00cm^3 of NaOH solution of unknown concentration was transferred to the titration flask by using pipette. 1-2 drops of phenolphthalein in indicator were added to the titration flask. With KHP solution was titrated until the end point was reached. Then the experiment was repeated three times.

Results:-

Concentration of KHP= 1.0×10^{-2} mol dm^{-3}

volume of NaOH solution= 25.00cm^3

Trial Number	Initial burette reading (cm ³) ±0.05 cm ³	Final Burette reading (cm ³) ±0.05 cm ³	Volume transferred (cm ³) ±0.05 cm ³
1	0.00	22.10	22.10
2	0.00	23.10	23.10
3	0.00	23.15	23.15

Calculation:-

Calculation mass of potassium hydrogen phthalate,

KHP concentration = KHP molar/ KHP volume

$$1.0 \times 10^{-2} \text{ mol dm}^{-3} = \text{KHP molar} / 250.00 \times 10^{-3} \text{ dm}^{-3}$$

$$\begin{aligned} \text{KHP molar} &= 1.0 \times 10^{-2} \times 250.00 \times 10^{-3} \\ &= 2.5 \times 10^{-3} \text{ molar} \end{aligned}$$

$$\begin{aligned} \text{KHP mass} &= \text{KHP molar} \times \text{KHP molar mass} \\ &= 2.5 \times 10^{-3} \text{ mol} \times 204.22 \text{ g mol}^{-1} \\ &= 0.51 \text{ g} \end{aligned}$$

Concentration of NaOH solution can be calculated by using the equation.



Due to equal molar ratio of KHP and NaOH,

Concentration = molar (C) / volume (V)

$$\begin{aligned} \text{KHP molar} &= \text{NaOH molar} \\ C_1 V_1 &= C_2 V_2 \end{aligned}$$

Where, C1 and V1 are the concentration and volume of the KHP and C2 and V2 are the concentration and volume of the sodium hydroxide acid.

Due to KHP concentration is $1.0 \times 10^{-2} \text{ mol dm}^{-3}$ and sodium hydroxide volume is 25.00 cm^3 ,

1 trial,

KHP volume= 22.10cm³

Moles = Concentration (C) * Volume (V)

Due to equal molar ratio of KHP and NaOH 1:1

Amount of KHP molar = Amount of NaOH molar

Concentration of KHP (C₁) * Volume of KHP (V₁) = Concentration of NaOH (C₂) * Volume of NaOH (V₂)

$$1.0 \times 10^{-2} \text{ mol dm}^{-3} * 22.10 * 10^{-3} \text{ mol dm}^{-3} = C_2 * 25.00 * 10^{-3} \text{ mol dm}^{-3}$$

$$\text{Concentration of NaOH} = \frac{1.0 * 10^{-2} \text{ mol dm}^{-3} * 22.10 * 10^{-3} \text{ mol dm}^{-3}}{25.00 * 10^{-3} \text{ mol dm}^{-3}}$$

$$= 0.88 * 10^{-2} \text{ mol dm}^{-3}$$

$$= 8.8 * 10^{-3} \text{ mol dm}^{-3}$$

2 trial,

KHP volume= 23.10cm³

Moles = Concentration (C) * Volume (V)

Due to equal molar ratio of KHP and NaOH 1:1

Amount of KHP molar = Amount of NaOH molar

Concentration of KHP (C₁) * Volume of KHP (V₁) = Concentration of NaOH (C₂) * Volume of NaOH (V₂)

$$1.0 \times 10^{-2} \text{ mol dm}^{-3} * 23.10 * 10^{-3} \text{ mol dm}^{-3} = C_2 * 25.00 * 10^{-3} \text{ mol dm}^{-3}$$

$$\text{Concentration of NaOH} = \frac{1.0 * 10^{-2} \text{ mol dm}^{-3} * 23.10 * 10^{-3} \text{ mol dm}^{-3}}{25.00 * 10^{-3} \text{ mol dm}^{-3}}$$

$$= 0.92 * 10^{-2} \text{ mol dm}^{-3}$$

$$= 9.2 * 10^{-3} \text{ mol dm}^{-3}$$

3 trial,

KHP volume= 23.15cm³

Moles = Concentration (C) * Volume (V)

Due to equal molar ratio of KHP and NaOH 1:1

Amount of KHP molar = Amount of NaOH molar

Concentration of KHP (C₁) * Volume of KHP (V₁) = Concentration of NaOH (C₂) * Volume of NaOH (V₂)

$$1.0 \times 10^{-2} \text{ mol dm}^{-3} \times 23.15 \times 10^{-3} \text{ mol dm}^{-3} = C_2 \times 25.00 \times 10^{-3} \text{ mol dm}^{-3}$$

$$\begin{aligned} \text{Concentration of NaOH} &= \frac{1.0 \times 10^{-2} \text{ mol dm}^{-3} \times 23.15 \times 10^{-3} \text{ mol dm}^{-3}}{25.00 \times 10^{-3} \text{ mol dm}^{-3}} \\ &= 0.93 \times 10^{-2} \text{ mol dm}^{-3} \\ &= 9.3 \times 10^{-3} \text{ mol dm}^{-3} \end{aligned}$$

In here, 1 trial's volume transferred and concentration can ignore. so, the second and third trials can be considered as final.

The average of the calculation is found by adding one and two and they dividing by two.

$$\begin{aligned} \text{Average of concentration of NaOH} &= C_2 \text{ trial 2} + C_3 \text{ trial 3} \\ &= \frac{(9.2 \times 10^{-3} \text{ mol}^3 \text{ mol dm}^{-3} + 9.3 \times 10^{-3} \text{ mol}^3 \text{ mol dm}^{-3})}{2} \\ &= 9.25 \times 10^{-3} \text{ mol dm}^{-3} \\ &= 9.3 \times 10^{-3} \text{ mol dm}^{-3} \end{aligned}$$

Difference between the experimental and actual concentration of sodium hydroxide,

= Average concentration – actual concentration

$$= 0.93 \times 10^{-2} \text{ mol dm}^{-3} - 1.0 \times 10^{-2} \text{ mol dm}^{-3}$$

$$= -0.07 \times 10^{-2} \text{ mol dm}^{-3}$$

$$= -7.0 \times 10^{-4} \text{ mol dm}^{-3}$$

Standard deviation of the result,

$$\begin{aligned} S_x &= \left(\left(\sum_{i=1}^n (x_i - \bar{x})^2 \right) / (n-1) \right)^{1/2} \\ &= \left[\frac{(2 \text{ trial con.} - \text{average con.})^2 + (3 \text{ trial con.} - \text{average con.})^2}{2-1} \right]^{1/2} \end{aligned}$$

$$\begin{aligned}
 &= \left[\frac{(0.92 \times 10^{-2} \text{ mol dm}^{-3} - 0.93 \times 10^{-2} \text{ mol dm}^{-3})^2 + (0.93 \times 10^{-2} \text{ mol dm}^{-3} - 0.93 \times 10^{-2} \text{ mol dm}^{-3})^2}{2} \right]^{1/2} \\
 &= \left[1 \times 10^{-8} \text{ mol dm}^{-3} \right]^{1/2} \\
 &= 1 \times 10^{-4} \text{ mol dm}^{-3}
 \end{aligned}$$

Error the average of concentration,

$$\% \text{ Error} = \left[\frac{\text{theoretical value} - \text{experimental value}}{\text{Theoretical value}} \right] * 100\%$$

$$\% \text{ Error} = \left[\frac{1.0 \times 10^{-2} \text{ mol dm}^{-3} - 9.3 \times 10^{-3} \text{ mol dm}^{-3}}{1.0 \times 10^{-2} \text{ mol dm}^{-3}} \right] * 100\%$$

$$= 7\%$$

discussion:-

Titration is important in chemistry as it allows for an accurate determination of solution concentration of the analyte.

During the course of the titration, the titrate (HCl) was added slowly to the NaOH solution. As it was added, the NaOH was slowly reacted away. If we added quickly we cannot accuracy end point.

Titration is a practical technique used to determine the amount or concentration of a substance in a sample. This concentration can then be calculated. To obtain valid results it is important that measurements are precise and accurate.

In here three trials were did to success the experiment. But in 1 trial, volume transferred is very different and less than 2 trial and 3 trial. In 1 trial, KHP has been expended 22.10cm³. but in 2 trial and 3 trial has been expended respectively 23.10cm³ and 23.15cm³. so here, if there is only ±0.05cm³ difference between the results of each trials, they included in the calculation to find concentration. So 1 trial result was not added to final calculation. 2 trial and 3 trial result were added to final calculation. So we separately calculated NaOH concentration each of 2 trial and 3 trial.

Finally two concentration of NaOH were received . Due to want final NaOH concentration, concentration of 2 trial and 3 trial were added together and the answer divided by 2.

Actual concentration of NaOH is $1.0 \times 10^{-2} \text{ mol dm}^{-3}$. Experimental value was $9.3 \times 10^{-3} \text{ mol dm}^{-3}$. When compare the actual value and experimental value, difference was $-7.0 \times 10^{-4} \text{ mol dm}^{-3}$. So due to that value is very small, the concentration of the experiment is close to the actual value. So accuracy is acceptable in this experiment.

The standard deviation is directly related to precision of a procedure where low standard deviations are indicative of a high level of precision. So in this experiment, standard deviation was very low. The value was $1 \times 10^{-4} \text{ mol dm}^{-3}$. Due to that value was very small value and close to zero, precision of data is also high and this experiment is precise. So this experiment is successful.

So in here the correctly volume should be received of transferred in burette and should check the correctly calibration of the balance and should receive lower point it. Exactly we should stop, adding KHP to NaOH when end point or color change point.

A weak acid – strong base titration is performed using s phenolphthalein indicator. Phenolphthalein is chosen because it changes color in a pH range between 8.3-10. It appears pink basic solution and colorless in acidic solutions. In here color change is pink color to colorless.

In here when KHP was added from beaker to the volumetric flask so that it escapes along the glass rod. Because to prevent KHP spill. Then refill the beaker with water and pour into the flask. After water was filled until 250 cm^3 , volumetric flask should be inverted because to homogenize.

Due to the fact, that all burette are made of glass, it can absorb and remain water on the surface, because of the polarity of the glass and intermolecular forces and have to rinse the burette with a solution which must be filled in it, because distilled water change the concentration of the initial solution.

The limitation of the equipment was realized. Sufficient quantities were used of analytical and titration. The precision of the glassware was verified.

Conclusion:-

The purpose of this lab was to determine the concentration of a sodium hydroxide solution by titrating it with a standard solution of known concentration.

Reference:-

<https://www.cerritos.edu> › LabPDF

Web results

Sodium hydroxide solution of about 0.2 M is prepared

<https://www.google.com/url?sa=t&source=web&rct=j&url=https://www.reference.com/science/reaction-khp-naoh-33126e9676754044&ved=2ahUKEwizt8z6wlbzAhW6ILcAHeB1CUcQFnoECDgQAQ&usg=AOvVaw0swbvwABpm5VFNMx6nvA0e>

<https://www.google.com/search?q=khp+and+naoh+reaction+reference&og=khp+and+naoh+reaction+reference&aqs=chrome..69i57j33i160l2.9357j0j9&client=ms-android-lgu-kr&sourceid=chrome-mobile&ie=UTF-8#>

[https://www.google.com/imgres?imgurl=https%3A%2F%2Fchem.libretexts.org%2F%40api%2Fdeki%2Ffiles%2F273168%2FFigure9.13.png%3Frevision%3D1&imgrefurl=https%3A%2F%2Fchem.libretexts.org%2FCourses%2FBethuneCookman+University%2FB-CU%253A+CH-345+Quantitative+Analysis%2FBook%253A+Analytical+Chemistry+2.1+\(Harvey\)%2F09%253A+Titrimetric+Methods%2F9.02%253A+Acid%25E2%2580%2593Base+Titrations&tbid=AoVVA_6cuUYRXM&vet=12ahUKEwiOkve7wlbzAhWCxnMBHbnUCaUQMygHegUIARDwAQ..i&docid=9v3XTLeeoY82oM&w=1415&h=1263&q=ph%20and%20btb%20indicator%20curve&client=ms-android-lgu-kr&ved=2ahUKEwiOkve7wlbzAhWCxnMBHbnUCaUQMygHegUIARDwAQ](https://www.google.com/imgres?imgurl=https%3A%2F%2Fchem.libretexts.org%2F%40api%2Fdeki%2Ffiles%2F273168%2FFigure9.13.png%3Frevision%3D1&imgrefurl=https%3A%2F%2Fchem.libretexts.org%2FCourses%2FBethuneCookman+University%2FB-CU%253A+CH-345+Quantitative+Analysis%2FBook%253A+Analytical+Chemistry+2.1+(Harvey)%2F09%253A+Titrimetric+Methods%2F9.02%253A+Acid%25E2%2580%2593Base+Titrations&tbid=AoVVA_6cuUYRXM&vet=12ahUKEwiOkve7wlbzAhWCxnMBHbnUCaUQMygHegUIARDwAQ..i&docid=9v3XTLeeoY82oM&w=1415&h=1263&q=ph%20and%20btb%20indicator%20curve&client=ms-android-lgu-kr&ved=2ahUKEwiOkve7wlbzAhWCxnMBHbnUCaUQMygHegUIARDwAQ)