Prepartion of primary standard

Preparation of Primary Standard solutions and Standardizing Acid and Base solutions Objectives: The objective of this experiment is: To prepare two primary standard solutions, KHP and Na2CO3. To standardize a sodium hydroxide solution using the prepared primary standard KHP. To standardize a hydrochloric acid solution using the prepared primary standard Na2CO3. To calculate the concentration of an unknown acid or base. Introduction A primary standard is a standard that is accurate enough that it is not calibrated.

For a compound to be considered as a primary standard it should have several important characteristics, the most important of which are high purity, stability, low hygroscopicity, high solubility, and high molar mass. A primary standard solution is a solution of known concentration made from a primary standard. Primary standard solutions are used in determining the concentrations of other solutions to an extremely high accuracy. They are typically used in titrations and other analysis techniques as standardization solutions.

A secondary standard solution, such as HCl solution, is a solution which must be standardized first against a primary standard, but afterwards, it will be stable enough for titrimetric work (Titration). Titration involves the gradual addition of a solution of accurately known concentration (standard solution) to another solution of unknown concentration (or vice versa), until the chemical reaction is complete. Titrations are based on reactions which go to completion rapidly. A reaction is complete when stoichiometric amounts of the reacting substances are combined.

This is the stoichiometric point (equivalence point) in the titration. The equivalence point is detected visually using an indicator. An indicator is a substance (added at the beginning of the titration to the flask) that changes color at (or very near) the equivalence point. The point where the indicator actually changes color is called the end point of the titration. In this experiment, two primary standards will be used. The first is potassium hydrogen phthalate (KHC8H4O4, abbreviated as KHP, molar mass = 204. 23 g/mol), an acid primary standard which will be used to standardize a sodium hydroxide solution.

The structure of KHP is shown below: O COH CO K O The chemical equation of the reaction can be written as: KHP(aq) + NaOH(aq) ? Or, expressed as a net ionic equation, HP-(aq) + OH-(aq) ? P2-(aq) + H2O(l) The second primary standard to be used is sodium carbonate, Na2CO3 (molar mass = 105. 99), a base, by which a hydrochloric acid solution will be standardized. The chemical equation of the reaction is: 2HCl(aq) + Na2CO3(aq) > CO2 (g) + 2NaCl(aq) + H2O(aq) KNaP(aq) + H2O(l) The reaction above generates CO2, which dissolves into the solution to generate an acid.

The presence of dissolved CO2 thus interferes with the pH and the detection of the end point of the titration. However, the CO2 can be driven off by boiling the solution, enabling an accurate titration. Procedure I. Standardization of NaOH a. Preparation of the acid primary standard. Obtain a bottle containing ~2g of KHP and weigh it with the cap on the analytical balance. Record the mass in Table 2. Transfer the solid KHP to a 100. 0 mL volumetric flask using a funnel, re-stopper the bottle and weigh it. Record the mass in Table 2.

Rinse the funnel to wash any sticking solid using a washing bottle and add more distilled water into the volumetric flask to dissolve the KHP (1/2 its capacity). Swirl the flask; make sure to dissolve the solid completely. Add more water (2/3) and swirl again. Dilute to the mark carefully, stopper or cover with a parafilm paper and invert several times with swirling to homogenize the KHP solution. b. Preparation of an approximately 0. 1 M NaOH solution 1. Obtain about 6 mL of a 50 % (w/v) NaOH solution in a clean and dry graduated cylinder from the stockroom. Transfer the NaOH to a clean 1L polyethylene bottle.

Fill the rest of the polyethylene bottle with double distilled water to the mark. Shake thoroughly to homogenize. Rinse your buret, after washing it with distilled water, with few mL of the NaOH solution; allow some solution to flow out through the lower end. Fill the rinsed buret with NaOH, make sure that the tip is filled with no air bubbles. c. Standardization of NaOH Pipet a 10. 00 mL aliquot of the primary standard KHP solution into a 125 mL Erlenmeyer flask. Add 25 mL of distilled water and two drops of phenolphthalein indicator. Record the buret reading (use a white card as background to facilitate reading the buret).

Put a white tile or paper below the Erlenmeyer flask and start titrating by adding NaOH continuously and with constant swirling of the flask. A pink color appears locally and disappears on swirling; continue titration till a faint pink color persists. Take the lower reading of the buret. The first titration is usually a rapid one. Repeat the titration slowly three more times. Record data in Table 2. Calculate the average molarity.

Standardization of HCl a. Preparation of the base primary standard 1. Obtain a bottle containing ~1g of dry Na2CO3 and weigh it with the cap on the analytical balance.

Record the mass in Table 2.

Transfer the solid Na2CO3 to a 100 mL volumetric flask using a funnel, re-stopper the bottle and weigh it. Record the mass in Table 2.

Rinse the funnel to wash any sticking solid using a washing bottle and add more distilled water into the volumetric flask to dissolve the Na2CO3 (1/2 its capacity). Swirl the flask; make sure to dissolve the solid completely. Add more water (2/3) and swirl again. Dilute to the mark carefully, stopper or cover with a parafilm paper and invert several times with swirling to homogenize the solution.

Standardization of HCl Get around 200 mL of HCl solution using a beaker and cover with a watch glass. Rinse your buret, after washing it with distilled water, with few mL of the HCl solution; allow some solution to flow out through the lower end. Fill the rinsed buret with HCl, make sure that the tip is filled with no air bubbles. Pipet a 10. 00 mL aliquot of the primary standard Na2CO3 solution into a 125 mL Erlenmeyer flask. Add 25 mL of distilled water and two drops of bromocresol green indicator. Record the buret reading (use a white card as background to facilitate reading the buret).

Put a white tile below the Erlenmeyer flask and start titrating by adding HCl continuously and with constant swirling of the flask until a change of color from blue to faint green. Boil the solution to expel CO2. The color should return to blue. Carefully add HCl from the buret until the solution turns green

again and report the volume of acid at this point. Keep the solution as reference for color for the other titrations.

Repeat the titration slowly three more times. Record data in Table 2.

Titration of blank Add to a 125 mL Erlenmeyer flask 50 mL of distilled water and two drops of bromocresol green ndicator. Titrate with your HCl solution to faint green. Subtract the volume of HCl needed for the blank from that required to titrate Na2CO3. Calculate the mean HCl molarity.

Determining the concentration of an unknown a. Qualitative identification of the unknown. Obtain an unknown from the stockroom and record its number in the report. Add two drops of the phenolphthalein indicator. Identify if it is an acidic or a basic unknown. Record yourobservation. Titration of the acidity in an unknown acid solution. Fill your burette with either HCl or NaOH according to your observation in the previous part. .

Pipet a 25 mL aliquot of the unknown solution into a 125 mL Erlenmeyer flask. Add two drops of the needed indicator (either phenolphthalein or bromocresol green). Record the buret reading (use a white card as background to facilitate reading the buret). Put a white tile or paper below the Erlenmeyer flask and start titrating by adding HCl or NaOH continuously and with constant swirling of the flask until a change of color according to the indicator being used. Record the lower reading of the buret. (Note: if your unknown is a base, remember to boil the solution as in part II. before proceeding with the titration to the end) 3. Repeat the titration slowly three more times. Record data in Table 2.

Reference:

1. Harris, Quantitative Chemical Analysis, 7th Ed. Student