Chapter 2: acid base titrations

1. Standardization of an aqueous solution of sodium hydroxide with potassium hydrogen phthalate

Any chemist is bound to meet the problem of carbonation of aqueous solution of sodium hydroxide. **In this practical work, you will have to standardize two solutions, which have not been stored in the same way.**

Sodium hydroxide solutions are widely used in laboratories. The problem of their expiry date is crucial for the quality of the analyses.

Two solutions of sodium hydroxide with concentrations close to 0.18 mol.L-1 are tested:

• Solution A: prepared a week ago and left open

• Solution B: prepared the day before

Their exact concentration obtained by standardization on the day of their preparation is indicated.

You are asked to examine whether the concentrations have evolved. In order to do this, you will carry out a titration by weighing a standard substance, potassium hydrogen phthalate. The end-of-reaction indicator is thymol blue.

The study will be conducted in pairs, one performing the calibration of solution A, the other that of solution B. Once you have finished your experimental work, combine your results and report as one conclusion.

**DOCUMENT 1 : Safety Information Sheet**

This information must be read BEFORE the experiment is attempted.

You are then required to sign the declaration at the bottom of the sheet and have this countersigned by a demonstrator or member of staff BEFORE commencing the practical.

DO NOT SIGN this form until you are satisfied that you appreciate the hazards associated with all aspects of this experiment.

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| Sodium Hydroxide Solution 0,25 mol.L-1 (From Sigma-Aldrich) |
| Potassium Hydrogen Phtalate 🡪 See the Safety Data Sheet |
| Phenolphtalein 🡪 See the Safety Data Sheet |

I have read the experimental procedure and the hazards assessments and will comply with these procedures.

Student : ………………………………………………………. Date : ………………………………

Demonstrator : …………………………………………………….

**DOCUMENT 2 : lab protocol**

1. **Introduction**

Primary Standards

In carrying out a titration, for example an acid-base titration, a solution of unknown strength is titrated against a standard solution whose concentration is known.

If you are carrying out a titration with a base such as sodium hydroxide you can obtain this solution from one of the containers in the lab. The container shows the concentration (molarity) of sodium hydroxide on the label. But how has this concentration been obtained? In the case of sodium hydroxide, solid sodium hydroxide pellets have been weighed out and dissolved in a known volume of water. However, sodium hydroxide is very hygroscopic (water absorbing) and so the pellets will absorb water during the weighing process. This results in an inaccurate value for the mass of sodium hydroxide as it is contaminated with a variable amount of water. Sodium hydroxide solution also absorbs carbon dioxide from the atmosphere (this is also an acid-base reaction). The carbon dioxide reacts to form sodium carbonate. We therefore say that sodium hydroxide is NOT a primary standard, and its concentration cannot be obtained accurately by weighing the solid and dissolving to a known volume.

A primary standard is a stable, non-hygroscopic, pure solid material, which can be weighed out and dissolved in water to give a solution of accurately known concentration.

A very useful primary standard is potassium hydrogen phthalate (MW = 204,22 g.mol-1):



Potassium hydrogen phthalate is a monoprotic acid derived from phtalic acid. Monoprotic means the acid has one replaceable hydrogen atom. It is a stable, non-hygroscopic, pure white solid which can be readily made up as a standard solution. It has a fairly high molar mass (hence reducing weighing error), but rather low solubility in water (<0,5 M at 25°C).

1. **Aims of the Experiment**

The aims of this experiment are:

1. To be familiar with what a primary standard is;
2. To remind you the techniques employed in carrying out a titration;
3. To have practice in calculating the result of a titration;
4. To get a realistic estimate of the errors associated with your standardization by comparing the results for the rest of the class;
5. To conclude about the preservation of sodium hydroxide solutions.
6. **Experimental Procedure**

Weigh out **accurately** (i.e. to 4 decimal places) between 0.3 and 0.4 g of solid potassium hydrogen phthalate into a weighing dish. Make a note of the accurate mass taken in your notebook. Transfer the solid to an erlenmeyer flask, washing any solid which remains on the boat into the flask. Dissolve the solid in the suitable volume of distilled water.

This solution will be used to standardize the approximately 0.18 mol.L-1 aqueous solution of sodium hydroxide provided in the laboratory.

Collect some of the sodium hydroxide solution provided in a beaker and transfer to a 25.0 cm3 burette. Run down the level of the solution so that the whole of the tube below the tap is filled with solution.

Add a few (2-3) drops of indicator to the solution of hydrogen phtalate and place the flask on a white tile. First carry out one rough titration adding the sodium hydroxide solution until the end-point is reached. The end point is when the solution’s color is persistent. Next, carry out at least two accurate titrations. In the accurate titrations run in the sodium hydroxide solution fairly rapidly until you are within 1-2 cm3 of the

end point determined in the rough titration. Then add sodium hydroxide solution one drop at a time until the first permanent color is obtained. Read the burette as accurately as possible to the nearest 0,05 cm3. Repeat the titrations in accordance to the ISO 5725-6 standard.

Write a report of your experiment in your notebook, including any observations.

1. **Calculations**

The reaction taking place in this titration is an acid-base neutralization reaction and the equation for the reaction is:



1. Find the expression of the number of moles of potassium hydrogen phthalate used.

Now find the molarity of the sodium hydroxide solution:

1. How many moles of sodium hydroxide solution react with potassium hydrogen phthalate to reach the end point of the titration (give only the expression) ?
2. Calculate the concentration of the sodium hydroxide solution and give the result

Analysis of the results:

1. Calculate the difference between your result and the value obtained by calibrating the same solution on the day of its preparation.

$$Er\left(C\_{X}\right)=\frac{|C\_{X}^{day of its preparation}-C\_{X}^{your result}|}{C\_{X}^{day of its preparation}} with X=A or B$$

1. Compare with your teammate result.
2. Comment on the deviations obtained and offer an explanation for these.

Activity summary

What you must remember:

- color indicator

- equivalence point

Skills linked to the curriculum**:**

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| **Compétences** | **Capacités à maitriser** |
| APP | S’approprier un cahier des charges, un mode opératoire S’approprier l’ensemble des informations liées à la sécurité |
| ANA | Reconnaître expérimentalement et dans la description d'un protocole un indicateur coloré acido-basique. |
| REA | Réaliser un protocole de titrage colorimétrique. Calculer et exprimer un résultat |
| COM  | Formuler et argumenter des réponses structurées Formuler et présenter une conclusion Utiliser des notions d’anglais technique à l’écrit et à l’oral  |