Lesson 1: Standardisation

*Read through the lesson notes. You can write them out, print them or save them.

*Once you have tried to understand the lesson answer the questions that follow and self-evaluate your work by checking the answers.

Learning Intention

-Learn about the importance of standardising chemicals in a laboratory.

-Learn about primary standards.

Background

When an analytical chemist uses a certain chemical in the laboratory it is often essential that they know the exact concentration of that chemical. The concentration stated on a label may be inaccurate as over time there can be slight changes in the concentration of chemicals.

Standard solutions

A standard solution is one of accurately known concentration and it can be prepared directly from a solute if that solute is a primary standard. To be suitable as a primary standard, a substance must meet a number of requirements.

- It must have a high purity.
- It must be stable in air and in solution.
- It must be readily soluble in a solvent (normally water).
- It should have a reasonably large relative formula mass in order to minimise the uncertainty in the mass of substance weighed out.

Some common primary standards are outlined below.

Sodium carbonate → Na_2CO_3

Hydrated oxalic acid → $(COOH)_2.2H_2O$

Potassium hydrogen phthalate → $KH(C_8H_4O_4)$

Silver nitrate → $AgNO_3$

Potassium iodate → KIO_3

Potassium dichromate → $K_2Cr_2O_7$

*Sodium hydroxide is not a primary standard as it absorbs moisture from the air and dissolves in it to form a very concentrated solution. Furthermore, both solid sodium hydroxide and a solution of it react with carbon dioxide from the air. Consequently, it is unstable in air and so does not meet the requirements of a primary standard.

Preparing a standard solution

-The sample of a primary standard is weighed accurately approximately. Although this term seems like a contradiction it means that you are instructed to weigh out as close as possible to a certain mass but ensure that you record the actual mass given on the balance.

For example, "weigh accurately approximately 3.2g of oxalic acid".

In this case it would be perfectly acceptable for the pupil to weigh out 3.16g of oxalic acid. The important point is that the pupil records 3.16g in their workbook and that they use 3.16g in any further calculations.

-The process of weighing is often described as weighing by difference, e.g.

Mass of weighing boat = 1.02g

Mass of weighing boat + oxalic acid = 4.18g

Mass of oxalic acid = 3.16g (obtained from the difference)

Once weighed out, the primary standard (oxalic acid) is transferred to a small beaker. The weighing boat is washed out using distilled water from a wash bottle to ensure that all of the primary standard has been transferred to the small beaker. More distilled water is added to the small beaker and the primary standard is dissolved with the aid of stirring using a glass rod (not a spatula).

The solution is then transferred to a volumetric (standard) flask, e.g. $250cm^3$. The beaker is further washed out with distilled water and the washings are transferred to the standard flask.

Towards the end, distilled water is added using a dropping pipette until the level of solution reaches the graduation mark on the volumetric flask. The flask is then stoppered and inverted several times.

Calculation to check concentration of primary standard (oxalic acid)

n = m/GFM GFM hydrated oxalic acid $(COOH)_2.2H_2O$ = 126g

n = 3.16/126

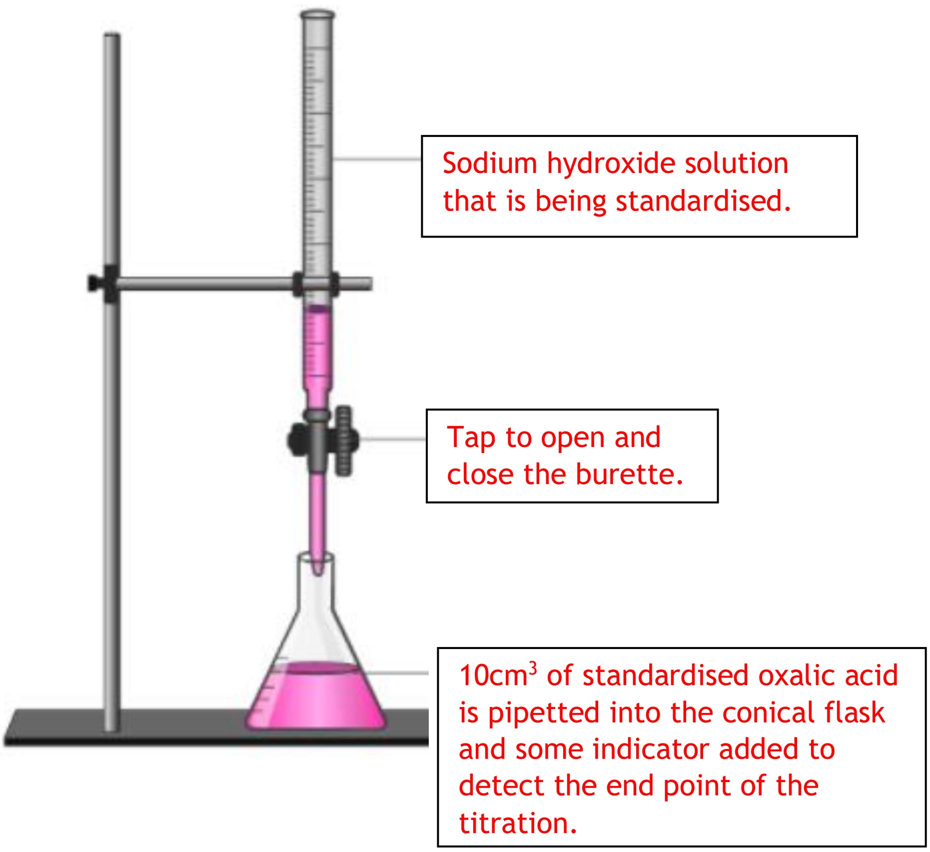
n = 0.025 moles

c = n/v

c = 0.025 / 0.25 (volume of volumetric flask)

c = 0.1 mol l^{-1}

In this case, the 0.1 mol l^{-1} oxalic acid solution can be used to standardise any common laboratory alkali, e.g. sodium hydroxide. This is carried out by means of a simple acid-base titration. The results of the titration can then be used to calculate the accurate concentration of the alkali.



Sample results and calculation

	Rough titration	First Titration	Second titration
Initial burette reading/cm^3	0	0	23.8
Final burette reading/cm^3	24.3	23.8	47.4
Total volume NaOH added/cm^3	24.3	23.8	23.6

average volume of sodium hydroxide =23.7cm^3

The rough titration is not considered as it is not concordant, i.e. +/- 0.2cm^3 of each other.

Equation for the reaction:

$2NaOH + (COOH)_2 \rightarrow Na_2(COO)_2 + 2H_2O$

Mole ratio

2 : 1

NaOH (COOH)$_2$

n = ? n =

c= ? c = 0.1 mol l^{-1}

v =0.0237 litres v = 0.01 litres

(average volume) (volume that is measured using a pipette)

moles of oxalic acid (n) = c x v 0.1 x 0.01 = 0.001 moles

oxalic acid : NaOH

1 mole : 2 mole

0.001 moles : 0.002 moles

concentration of NaOH = n/v 0.002/0.0237 = 0.084 mol l^{-1}

In this instance, it means that the concentration of the laboratory sodium hydroxide is 0.084 mol l^{-1}. Therefore, rather than using 0.1 mol l^{-1} (as would be indicated on the label) the more accurate concentration is used.

→Watch the clip on Youtube.

https://www.youtube.com/watch?v=iPYyRNjXkgY

→ Read Scholar Heriot-Watt/ Researching Chemistry Section 5.2.

→ Read Bright Red Advanced Higher Textbook Page 82.

→ Answer the questions from Sheet 4.1 and check the answers when you have completed them.

4.1 Standardisation

1. A pupil was asked to prepare a standard 0.1 mol l^{-1} solution of the primary standard potassium iodate (KIO_3 GFM 214g) to use in an experiment. They were asked to weigh accurately approximately 4.3g. The pupil was also asked to weigh by difference when preparing the sample of potassium iodate.

a) Explain what is meant by weigh accurately approximately in this context.

b) Explain how the pupil would weigh by difference.

c) Explain why potassium iodate is a suitable substance to be used as a primary standard.

2. In another experiment a pupil was asked to standardise approximate 0.1 mol l^{-1} hydrochloric acid using sodium carbonate (Na_2CO_3) as the primary standard.

Outline with as much detail as possible how the pupil would prepare 250 cm^3 of 0.1 mol l^{-1} of the primary standard and how this would be used to standardise the approximate 0.1 mol l^{-1} hydrochloric acid.

(in your answer you should include the suitable equipment that the pupil would use and include any measurements with values that they would make).

3. A student used a standard solution of 0.105 mol l^{-1} oxalic acid to standardise 25.0 cm^3 of an unknown concentration of potassium hydroxide solution. The equation for the reaction is

$$H_2C_2O_4(aq) + 2KOH(aq) \rightarrow K_2C_2O_4(aq) + 2H_2O(l)$$

The raw results for the titration are given in the table.

	1st attempt	2nd attempt	3rd attempt
Initial burette reading (cm^3)	0.0	20.6	0.1
Final burette reading (cm^3)	20.6	38.9	18.5
Titre (cm^3)	20.6	18.3	18.4

a) Explain why the volume of oxalic acid used for the calculation was taken as 18.35cm^3.

b) Calculate the concentration, in mol l^{-1}, of the potassium hydroxide solution.