Lesson 15: Use of Controls

*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 using controls as part of a chemical investigation.

Background

You will have gained a brief understanding about the importance of using controls in experiments. The use of controls is an important aspect of experimental design and forms a fundamental part of validating scientific research. This lesson will outline several examples of common controls that are carried out in Advanced Higher investigations.

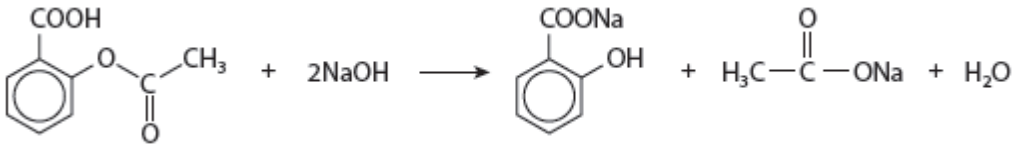
Use of controls in experimental work

Experimental design is an important part of research and forms a key step of the planning process when undertaking a project of practical work. When carrying out research and investigative work you want to ensure that your procedure is working and giving the desired result. One of the first tasks that you would do is to validate your procedure by using a control to show it produces a positive outcome. It is a logical step but one that can often be overlooked.

In this lesson, we will look at some familiar examples of experiments and controls that are used.

Example 1: Determination of aspirin in tablets

Previously, we learned that for the determination of aspirin in tablets a back titration has to be carried out. The aspirin tablets are first dissolved in sodium hydroxide:



The excess sodium hydroxide is then titrated against hydrochloric acid and working back this allows the mass of aspirin in the tablets to be determined

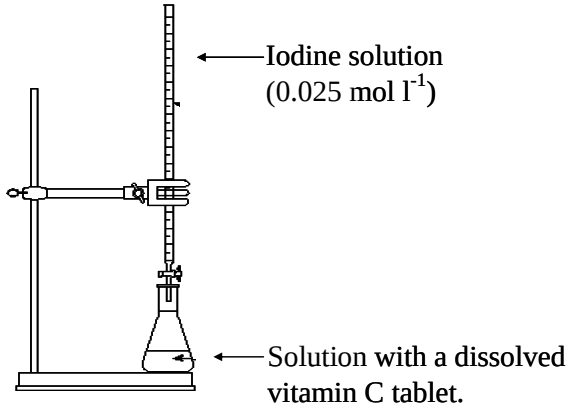
$$2NaOH + H_2SO_4 \rightarrow 2H_2O + Na_2SO_4$$

A suitable control that can validate this technique is to take a known mass of pure aspirin and treat it in exactly the same manner as described above. If the experimentally determined quantity of aspirin is very close to the known value then it can be concluded that the method is valid and therefore any results obtained will be reliable.

Example 2: Determination of vitamin C

For the determination of vitamin C in tablets or fruit juices a redox titration with iodine solution is carried out.

$C_6H_8O_6 + I_2 \rightarrow C_6H_6O_6 + 2H^+ + 2I^-$



A suitable control for this investigation could be to titrate a known concentration of ascorbic acid (vitamin C).

Example 3: Determination of ethanol content in alcoholic drinks

To investigate the ethanol content in alcoholic drinks, a back titration involving acidified dichromate solution is used. The reaction between the acidified dichromate solution and ethanol is given below.

$3C_2H_5OH(aq) + 2Cr_2O_7^{2-}(aq) + 16H^+(aq) \rightarrow 3CH_3COOH(aq) + 4Cr^{3+}(aq) + 11H_2O(\ell)$

A suitable control for this investigation is to treat a sample of ethanol of known concentration is the same manner.

Example 4 (Identification of an Unknown Organic Compound)

A common Advanced Higher Chemistry project and indeed undergraduate laboratory investigation is the determination of an unknown organic compound. The investigation consists of carrying out a series of experiments to work out the name and therefore structure of an unknown compound. Any number of experiments can be carried out and clearly the more positive results you obtain, the more likely you will be to name correctly the unknown compound.
An essential part of this investigation is to use a control for each experiment before testing the unknown compound.

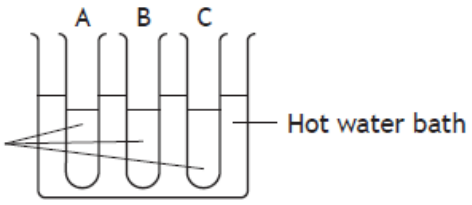
-*Testing for unsaturation*.
To determine whether the unknown organic compound contained a carbon to carbon double bond, you could test it with bromine solution. However, you would use a control such as hex-1-ene first to ensure that it rapidly decolourises the bromine solution. This would validate the experiment and ensure that the bromine solution was suitable for determining whether the unknown compound was unsaturated or saturated.

-*Distinguishing between an aldehyde and ketone*.
From Higher Chemistry you will have tested unknown compounds with oxidising agents to investigate which is an aldehyde and which is a ketone. Chemically, we know that aldehydes will be oxidised (and give a colour change with the oxidising agents) and ketones will not be oxidised (and give no colour change with the oxidising agents).

A simple but effective control would be to gently warm propanal in a water bath with the common oxidising agents:

Tollen's reagent → a silver mirror would be produced
Benedict's reagent → solution would change from blue to orange
Acidified potassium dichromate → solution would change from orange to blue/green

Propanal acting as a control by being gently warmed with three oxidising agents A, B and C. In each case a colour change would be observed.



-Presence of carbonyl functional group (using Brady's reagent)

Although no longer in the Advanced Higher Chemistry course, a useful experiment for detecting the presence of a carbonyl group is to use 2,4-dinitrophenylhydrazine (commonly known as Brady's reagent).
When Brady's reagent is mixed with an aldehyde or ketone (both contain a carbonyl group), an orange precipitate forms. In this case, a possible control would be to mix a sample of butanone with Brady's reagent and ensure that an orange precipitate forms.
This would then allow you to test the unknown organic compound with Brady's reagent and determine if it contains a carbonyl compound.

The above examples will have given you a brief introduction to using controls. The study and use of controls in scientific research is much more complex, however, this lesson will have provided you with some basic examples that you are familiar with from your prior knowledge.

TASK

→ Answer the questions from Sheet 4.15 and check the answers when you have completed them.

4.15 Use of Controls

1. 0.02 moles of the salt, $Pt(NH_3)xCl_y$ were dissolved in nitric acid and excess silver (I) nitrate solution was added. The precipitate formed was filtered, washed and dried. It weighed 5.74g.
   The number of moles of chloride ions per mole of the salt is

   A. 1
   B. 2
   C. 3
   D. 4

2. Which of the following aqueous solutions contains the greatest number of negatively charged ions?

   A. 500 cm^3 0.10 mol l^{-1} $Na_2SO_4(aq)$
   B. 250 cm^3 0.12 mol l^{-1} $BaCl_2$ (aq)
   C. 300 cm^3 0.15 mol l^{-1} KI(aq)
   D. 400 cm^3 0.10 mol l^{-1} $Zn(NO_3)_2(aq)$

3. In which of the following separation techniques is partition between two separate phases not a part of the process?

   A. Recrystallisation of benzoic acid from hot water
   B. Separation of alkanes using gas-liquid chromatography
   C. Separation of plant dyes using paper chromatography
   D. Solvent extraction of caffeine from an aqueous solution using dichloromethane

4. When a salt, formula $Ni(OH_2)_6.K_2(SO_4)_2$, is dissolved in water, the solution contains the ions $Ni(OH_2)_6^{2+}$, K^+ and SO_4^{2-}.
   The total number of moles of ions in one litre of 0·01 mol l^{-1} solution is

   A. 0·01
   B. 0·03
   C. 0·05
   D. 0·10.

5. EDTA forms a 1:1 complex with Ni^{2+} (aq). What is the concentration, in mol l^{-1} of a nickel (II) solution, if 20cm^3 of it reacts with $2x10^{-3}$ moles of EDTA?

   A. 0.002
   B. 0.01
   C. 0.02
   D. 0.1

6. The reaction between copper metal and silver(I) nitrate solution can be written as follows.

   $Cu(s) + 2AgNO_3(aq) \rightarrow Cu(NO_3)_2(aq) + 2Ag(s)$

   The addition of excess copper metal to $100cm^3$ of silver(I) nitrate solution produces a precipitate of 0.108g of silver metal.
   What quantity of silver(I) nitrate solid was used to make up the $100cm^3$ of silver(I) nitrate solution?

   A. 0.002 moles
   B. 0.34g
   C. 0.001 moles
   D. 0.017g

7. $2Cu^{2+}(aq) + 4I^-(aq) \rightarrow 2CuI(s) + I_2(s)$
   $2S_2O_3^{2-}(aq) + I_2(s) \rightarrow S_4O_6^{2-}(aq) + 2I^-(aq)$

   $50cm^3$ of 0.02 mol l^{-1} $CuSO_4$ solution are added to excess KI solution. What volume of 0.10 mol^{-1} $Na_2S_2O_3$ is required to react completely with liberated iodine?

   A. $5cm^3$
   B. $10cm^3$
   C. $25cm^3$
   D. $50cm^3$

8. Solid ammonium dichromate decomposes to produce chromium (III) oxide, nitrogen and water. The complete decomposition of one mole of ammonium dichromate would give

   A. a total of one mole of all the products
   B. 3.0 moles of water
   C. 2.0 moles of chromium (III) oxide
   D. 1.0 mole of nitrogen

9. Which of the following is an acceptable analytical method?

   A. Determination of the concentration of Ca^{2+} ions by titration with acidified potassium permanganate.
   B. Determination of the concentration of Ni^{2+} ions by titration with EDTA
   C. Determination of the concentration of NO_3^- ions using barium chloride
   D. The use of solid sodium hydroxide to standardise hydrochloric acid.

10. The oxidation of ethanedioate ions ($C_2O4_2^-$) in aqueous solution can be represented by the ion-electron equation:

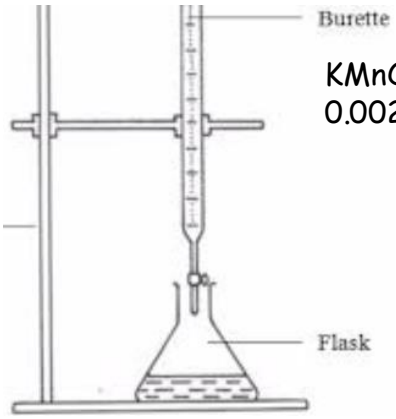
    $C_2O_4^{2-}(aq) \rightarrow 2CO_2(g) + 2e$

    What volume, in cm^3, of acidified 0.10 mol l^{-1} potassium permanganate solution is required to oxidise completely a solution containing 0.01 mole of ethanedioate ions?

    A. 25
    B. 40
    C. 80
    D. 100

11. The analysis of iron content in ferrous sulfate tablets can be carried out by titrating a solution of iron tablets with acidified potassium permanganate.
    Two iron tablets were crushed up and dissolved in small volume of dilute sulfuric acid. This was then added to a $100cm^3$ volumetric flask with the washings. Distilled water was then added to the flask until the graduated mark. The flask was inverted several times.
    10 cm^3 samples of the iron tablet solution were titrated against potassium permanganate. The average titre was $24.75cm^3$.

    $5Fe^{2+} + MnO_4^- + 8H^+ \rightarrow Mn^{2+} + 5Fe^{3+} + 4H_2O$



$KMnO_4^-$
0.0020 mol l^{-1}

10 cm^3
Iron tablet
solution

    a) Why is no indicator required for this analysis?

    b) Calculate the mass, in mg, of iron in one tablet.

    c) Describe a suitable control experiment that could be used to validate this technique.