RSC/AD Western Region

Schools' Analyst Competition

Bristol

Analytical Competition –Theoretical Task 2017

Safety: There is no equipment or chemicals for this task-however you must still wear lab coats and safety spectacles as you are in the lab. Gloves for this are optional.

The Task

You are presented with 5 colourless solutions labelled A-E. The solutions are known to be 2.00 mol dm^{-3} sodium carbonate solution, 2 mol dm^{-3} hydrochloric acid, 0.5 mol dm^{-3} hydrochloric acid, phenolphthalein solution and 0.5 mol dm^{-3} silver nitrate solution.

Using only these chemicals and test tubes explain how you identify each solution A-E. [10 marks]

[There is more than one way for some of these solutions- only one is needed]

Solution	**What you would do**	**What you would expect to see**
2 mol dm^{-3} sodium carbonate solution		
2 mol dm^{-3} hydrochloric acid		
0.5 mol dm^{-3} hydrochloric acid		
phenolphthalein solution		
0.5 mol dm^{-3} silver nitrate solution		
School name		

Iron Tablet Determination Analytical Competition

The analysis of iron tablets by titration using acidified potassium manganate(VII) solution.

Setting the Scene

Iron tablets contain iron(II) sulfate which is soluble in water.

The experiment is to determine the average percentage by mass of iron(II) sulfate in the tablets provided. You will use the method of titration to provide data to answer this question.

Iron(II) ions can be oxidised to iron(III) ions by acidified potassium manganate(VII) [$H^+/MnO_4^-{}_{(aq)}$]. In these conditions the purple colour of the solution of manganate(VII) ions is reduced to a very, very pale pink (almost colourless) solution of manganese(II) ions.

The manganate(VII) ion is reduced to colourless Mn^{2+} ions:

$MnO_4^- (aq) + 8H^+ (aq) + 5e^- \rightarrow Mn^{2+}(aq) + 4H_2O(l)$

The iron(II) ions are the reducing agent as they provide the electrons:

$Fe^{2+}(aq) \rightarrow Fe^{3+}(aq) + e^-$

The overall equation is therefore:

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

The potassium manganate(VII) solution is added from the burette to the solution of the acidified iron sulfate solutions and is immediately decolourised. Once the reducing agent is used up, the next drop of potassium manganate(VII) solution is not decolourised and colours the solution in the conical flask a pale purple colour. Prior to this swirling the mixture takes progressively longer to decolourise the initial colour formed at the surface. The end-point is the first appearance of this pale purple colour i.e. no indicator is required.

Here dilute sulfuric acid is used to provide $H^+(aq)$, which should always be in excess otherwise insoluble brown manganese(IV) oxide will form. The volume of acid you are told to use in the method will make sure that there is a large excess of sulfuric acid each time.

Health and Safety

At all times in the laboratory safety glasses/googles and lab coat should be worn. Disposable gloves should be worn at the work bench.
Broken glass- notify a technician or a judge straight away so that it can be cleaned away safely.
Spilt chemicals- notify a technician or a judge straight away so that it can be cleaned away safely.

Sulfuric acid (1 mol dm^{-3}) is corrosive- Wear safety glasses. If spilt on skin wash off with plenty of water. If in the eye irrigate with water. Seek medical attention.

Potassium manganate (VII) (0.020 mol dm^{-3}) solution is an irritant. Wear safety glasses. If spilt on skin wash off with plenty of water. If in the eye irrigate with water. Seek medical attention.

Iron tablets (Iron sulfate, ferrous sulfate) - an overdose may be fatal. Solutions are Irritants-wear safety glasses).

Method

a) Put your school name on a piece of A4 paper (on which all results and calculations will be recorded and handed in for marking)
b) Using a weighing boat or watchglass, weigh accurately five iron tablets. Record both the total mass and the number of tablets that you are using.
c) Weigh a clean pestle and mortar. Record.
d) Grind up the tablets you have weighed out and add the powder to a 100 ml conical flask and add 50 cm^3 of the 1 mol dm^{-3} sulfuric acid. Stopper and shake for 5 minutes (timed) to dissolve the iron sulfate. Weigh the used pestle and mortar and calculate the mass of tablets added to the conical flask. Record both.
e) Some iron tablets have an insoluble outer coating. The solution will now need to be filtered into a 100 cm^3 volumetric flask.
f) Rinse the residue in the filter paper into the graduated flask using a small volume of de-ionised or distilled water.
g) Add dilute sulfuric acid to make the solution in the graduated flask up to the mark, stopper and shake to make homogeneous.
h) Construct a table into which you can record your measurements and volume calculations for the titration.
i) Rinse and fill the burette with the 0.0200 mol dm^{-3} potassium manganate(VII) solution.
j) Pour some of the contents of the graduated flask into a clean, dry 250 cm^3 beaker. Use a 25 cm^3 pipette and a pipette filler, measure out a 25.0 cm^3 sample of the iron(II) sulfate solution into a clean 250 cm^3 conical flask. IF you don't know how to use this type of pipette filler ask a judge for some guidance - you will not be penalised.
k) Using a 25 cm^3 measuring cylinder, measure out 25 cm^3 of the 1 mol dm^{-3} sulfuric acid provided and add this to the contents of the conical flask. Titrate this acidified sample of iron(II) sulfate solution by adding potassium manganate(VII) from the burette until the first permanent pink colour is seen after swirling.

l) You will only be able to carry out three titrations. You should be able to obtain at least two, if not 3, results that are concordant. Record all three results that you obtain. Calculate and record the mean volume of potassium manganate(VII) solution used in the titration (the average (mean) titre). Indicate whether you have used any anomalous result(s).
m) Calculate the number of moles of manganate ions you used, on average mean), in your titration.
n) Using the balanced equation calculate how many moles of iron(II) ions were involved in the titration.
o) Calculate how many moles of iron(II) ions were in the 5 ground up tablets used. Hence calculate how many moles of iron(II) ions were in the unground tablets.
p) Last, calculate the average mass of iron ions in the iron tablets you were given.
q) Show all you workings (made clear) on the sheet you are to hand in.

Rates of Reaction/ Cascade Task

Introduction

The iodine clock reaction is a favoured reaction of many chemistry teachers. In this reaction a mixture of, ethanoic acid, potassium iodide, starch and hydrogen peroxide solution react in a two stage reaction to eventually produce iodine, which in the presence of starch turns blue-black. The initial reaction is slow followed by a faster second step liberating iodine. Knowledge of the actual chemistry is not needed here.

There are several variations on the reaction instructions.

You are going to be presented with Solution A (containing all the reactants bar the hydrogen peroxide) and a 5% solution of hydrogen peroxide solution. There will be two tasks:

Task 1 You will make dilutions of the hydrogen peroxide solution and look at the effect on the time of reaction. These results will be graphed.

Task 2 You will use the graph of your results to make up three solution mixtures that will change colour 10 seconds after an initial reaction has changed colour.

You will be judged specifically on safe working, tabulation of results, your graph and how close your reactions are to the time intervals stated (as judged by a judge who will be doing the timing.

Safety

Wear safety glasses, lab coat and rubber gloves correctly when performing this task.

Solution A

This contains a dilute mixture of potassium iodide, very dilute ethanoic (acetic) acid and starch.

Slightly hazardous in case of eye contact (irritant), of ingestion. Non-corrosive for skin. Non-sensitizer for skin. Non-permeator by skin.

First Aid

Eye Contact: Immediately flush eyes with running water for at least 15 minutes, keeping eyelids open. Cold water may be used.
Skin Contact: No known effect on skin contact, rinse with water for a few minutes.
Serious Skin Contact: Not available.
Inhalation: Allow the victim to rest in a well-ventilated area. Seek immediate medical attention

5% Hydrogen peroxide Solution

Eyes: May cause irritation, redness, pain, and tearing.

Skin: May cause irritation and bleaching of the hair and skin.

Ingestion: May cause irritation and burning of the lips, mouth, and throat, painful swallowing and nausea.

Inhalation: May cause irritation of the respiratory tract.
First Aid

Eyes: Immediately flush eyes with water for at least 15 minutes. Immediately get medical assistance.
Skin: Flush with water for 15 minutes. Get medical assistance if irritation develops.
Ingestion: DO NOT induce vomiting. Dilute with water or milk. Get medical assistance.

Source
http://fscimage.fishersci.com/cmsassets/downloads/segment/ScienceEducation/pdf/Chemicals/MSDS/S25361_HP1206.pdf

Method

Temperature agitation, volume and concentration all affect rates of reaction, Control all variables as best you can (a thermometer, hot water and an ice bath are available as needed). The reaction should be investigated at lab temperature. Washing up glassware in hot water may cause error unless the glass is cooled back to room temperature as will not drying washed up beakers.

Using whichever glassware you feel most appropriate from that provided measure out 20.0 cm3 of solution A and transfer to a clean, dry beaker. Add 20.0 cm^3 of the 5% hydrogen peroxide solution starting a stopwatch at the point you start pouring. Swirl the mixture 3 times and place on the bench. Stop the stopwatch once the colour appears. The colour should appear all at the same time else the swirling was inefficient at mixing. Record the time and repeat.

Using appropriate glassware dilute the 5% hydrogen peroxide and eventually make solutions that are 4%, 3.5%, 3% and 2%. You will need to make sufficient of each solution to run each concentration twice (ie a minimum of 40 cm3 of each), Run each against 20 cm3 of Solution A. Record your results in an appropriate table.

Plot a graph of concentration of hydrogen peroxide against average time (to the nearest 0.5 s).

Task 2: Using your graph choose two concentrations that will change colour 10 seconds apart, i.e. it does not matter when your first reaction changes but the second must change 10 seconds later (as judged by a judge). You will gain marks according to how close it is to the 10 seconds. (Note: 9.5 seconds is as good as 10.5 seconds).

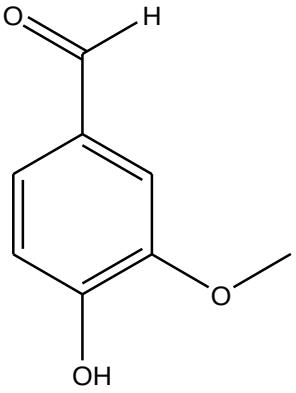
Your graph and table of results must be handed in for marking.

The Concentration of Vanillin in Vanilla Extract - **Sheet one**

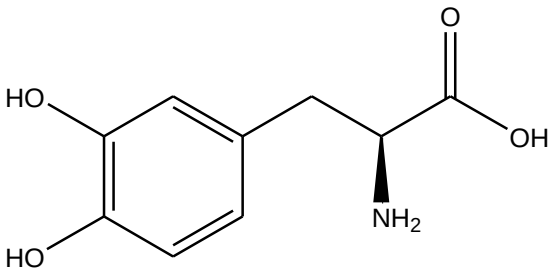
The following analytical chemistry experiment is based on an experiment 'The Determination of Vanillin in a Commercial Vanilla Extract' published by Eric W. Ainscough and Andrew M. Brodie in the Journal of Chemistry Education Volume 67 Number 12 December 1990

Vanillin is the chemical that gives vanilla its familiar flavour and smell. The common name for vanillin is 3-methoxy-4-hydroxy-benzaldehyde.
Vanillin can be extracted by ethanol from the beans of the vanillin plant – a member of the orchid family. Vanillin may also be obtained from lignin, a waste material from paper pulp production. It can also be synthesised from 2-methoxyphenol.
Vanillin is used as the starting material for several drugs such as L-dopa which is used in Parkinson's disease treatment.

Equations and structures



Vanillin



L-Dopa

This experiment contains elements of organic, inorganic and physical chemistry.

- Organic- a solvent extraction
- Inorganic- a salt preparation using involving NaOH
- Physical the use of ultraviolet spectroscopy

The experiment is in two parts: part one is the extraction of the vanillin and part two is the preparation of a calibration curve.

Safety information

Even though the wearing of appropriate clothing, (check if unsure what this means) lab coats, disposable gloves and safety glasses is a standard procedure you also need to know the potential risks of the chemicals being used in this experiment.

Each group member should read these before beginning the practical. Each member of the team should sign to say they have read and understood this.

Chloroform ($CHCl_3$, 1,1,1-trichloromethane)

Toxicology
This material causes cancer in laboratory animals, and is IARC listed as a probable human carcinogen. Inhalation and ingestion are harmful and may be fatal. May cause reproductive damage. Irritant. Exposure to alcohol may increase toxic effects. Prolonged or repeated skin contact may cause dermatitis. Typical TLV 50 ppm
Personal protection: Safety glasses and gloves. Good ventilation / use in fume cupboard

Sodium Hydroxide Solution (0.1mol dm^{-3} NaOH.)

Harmful in this dilution. Contact with the eyes can cause serious long-term damage. If you get in the eyes wash out for at least 10 minutes and get medical attention straight way.

Personal protection: Safety glasses and gloves.

Vanillin
Harmful if swallowed (ORL-RAT LD50 1580 mg kg^{-1}).
May be harmful by inhalation or in contact with skin. Eye irritant.

Keep this sheet beside your experiment

The Concentration of Vanillin in Vanilla Extract - Sheet two

Part 1: The Extraction of the Vanillin

Ask a demonstrator to show you how use the separating funnel or refer to the techniques manual before you begin.

Read all the way through before beginning this experiment

Carry out all handling of chloroform and chloroform stages in a fume cupboard.

Add 10 cm^3 of deionised or distilled water to a 50 cm^3 separating funnel.
Accurately pipette 1 cm^3 of the commercial vanilla extract into the separating funnel.
Add 20 cm^3 of chloroform (1,1,1-trichloromethane) to this mixture, stopper and shake well, releasing the pressure by turning the tap as required.

Clamp the separating funnel, remove the stopper and let the layers settle out.
Drain the lower chloroform layer into a 250 cm^3 separating funnel.
Repeat the extraction of the remaining aqueous layer with two more aliquots (portions) of chloroform. There should now be 60 cm^3 of chloroform containing the vanillin in the 250 cm^3 separating funnel. [Note: 3 extractions of 20 cm^3 will dissolve more vanillin than one 60 cm^3 extract!].

Carefully add 50 cm^3 of 0.1 mol dm^{-3} sodium hydroxide to the separating funnel and shake. The vanillin will react with NaOH and will move into the top aqueous layer.
Drain the chloroform layer into a beaker and run the aqueous layer into a 250 cm^3 volumetric flask.
Extract the chloroform with a further two 50 cm^3 portions of 0.1mol dm^{-3} NaOH.
Combine all the aqueous layers into the 250 cm^3 volumetric flask and make up to the mark with more 0.1mol dm^{-3} NaOH shaking to make the solution homogenous.

Discard chloroform to the waste organic solvents bottle (**NOT DOWN THE SINK!**).

Part 2: The creation of the calibration curve / line

You are provided with a standard vanillin solution that contains 100 micrograms per cubic centimetre (100µg cm^{-3}) in 0.1 mol dm^{-3} NaOH. From this solution you will need to prepare standard solutions of 5, 4, 2 and 1 µg cm^{-3} in the 50 cm^3 volumetric flasks provided. Carry out all the dilutions with 0.1mol dm^{-3} NaOH.

Measure the absorbance of your sample prepared in the extraction stage and the for standard vanillin solutions using the ultraviolet (uv) spectrophotometer. Having rinsed out your cuvette with the solution to be used first (making sure that the outside using dry using the tissues provided) pour each sample into a cuvette. The spectrophotometer should be set to a wavelength of 347nm. Record a table of absorbances with concentration.

Plot a graph of absorbance versus concentration of your standard solutions. This is the calibration curve/line.

Hence deduce the concentration of the unknown vanillin concentration.

Questions :

1. Why does the characteristic smell of vanillin disappear when it is dissolved in the sodium hydroxide?
2. How could you check that there was no vanillin left in the chloroform layer?
3. What does ‘nm’ stand for?