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We acknowledge the grateful assistance of Lawson Cockcroft for his inspirational work in using Vernier data logging system in the teaching of chemistry. Lawson Cockcroft, a few biographical details: 10 years teaching chemistry at grammar school, GCSE and A Level, then the rest of the time teaching Ordinary National and Higher National Certificate Chemistry to part-time day release students at technical college, eventually ending up teaching Physical Chemistry to GRSC and honours degree level. Now retired but still involved with the Chilterns & Middlesex Section of the Royal Society of Chemistry and is now mainly interested in applying ICT to practical work and individual assignments.



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LoggerPro Features

File Menu

The **Print** option results in the full screen being printed. (There is a draw-back to using this when there is a table – with Hewlett Packard printers, the shaded border prints black and obscures the headings of the columns). To print just the graph or data table, select **Print Graph** or **Print Table** respectively.

The **Preferences** option displays the **Applications Preferences** .

the **Default Folder** can be changed from the Vernier Software folder to any other option

the **Curve Fit** default setting (**Allow Automatic Curve Fit**) can be changed to allow manual curve fitting.

the **Default Precision** can be changed from the default (4) to any value in the 1 – 9 range, with the additional option of **Decimal Places** or **Significant Figures**

the available **Presentation** options are to **Display Larger Text on Screen** and to draw **Thick Graph Trace Lines**

the number of **Undo Levels** can be changed from the default (16) within the range 0 to 64

The **Settings For Filename** options are

angle units for trig calculations may be Radians (the default) or degrees

the number of points used in calculating derivatives (default is 5) may be altered within the range 3 – 97

the number of points used in the smoothing calculation (default is 5) may be altered within the range 3 - 97

Data Menu

The **New Calculated Column** allows a calculation to be performed on the data being collected. The **New Calculated Column B** window has three tabs –

Column Definition entry boxes are provided for the column name, a short name (as a graph/column label when the name is too long for the space available) and the units. An Insert symbol box allows a degree ($^{\circ}$) and a micro (μ) symbol to be inserted. A further set of boxes allows the equation defining the calculation to be entered, displays the available pre-set functions and allows the variables to be inserted into the equation.

Options the **Point Protectors** allow changes to be made to

Style	to be selected from a range of different patterns
Display Every	the default setting is 1 (every point displayed)
Use Column	sets the column to be used
Error Bar Calculation	can be set as either a percentage or fixed value

Column Options this allows the above calculated column options to be applied to the original columns

Analyse Menu

The **Replay** option displays a **Replay** window which allows the plotted data to be re-run at the original speed or at a faster or slower speed set by a slider bar.

Creating Logger Pro Data Logging Files

When Logger Pro is started without the interface being attached to the computer, a box appears offering the options of attaching the interface or proceeding without it. The following notes refer to working without sensors attached – this is how you would proceed if you were setting out to plan a data logging experiment. When a sensor is attached to the interface, the corresponding table and graph are displayed automatically and you would start from step 2 in each of the procedures described below.

Basic Data Logging

1 Continuous recording of data.

The first step is to click on **Experiments** and select **Show Sensors**. This displays a Sensors(Lab Pro) window, listing icons of all the available sensors. Drag the appropriate sensor to the appropriate channel (CH1 – CH4 for the analogue sensors on the left of the window, DIG/SONIC for the digital sensors on the right of the window). After attaching the sensor(s), click on the **Close** button. This results in a data table and graph appropriate to the sensor being displayed. The data table is headed **Latest**, with Time as the X variable and the property measured by the sensor as the Y variable.

The second step is to click on **Experiments** to set up the data collection. From the drop-down menu, select the **Data Collection** option. This displays a **Data Collection** window with two tabs, labelled **Collection** and **Triggering**, respectively. The default setting is to collect data in a continuous stream (**Time Based** mode). The duration of the data collection is set by entering the appropriate number and time unit in to the **Length** boxes (time units available are milliseconds, seconds, minutes and hours). The sampling rate is set by moving a slider bar until the appropriate samples/second or seconds/sample is

shown or by entering a number directly into the box. (For oversampling, see later). Click the **Done** button to return to the screen.

This completes the setting-up procedure and the file can then be saved in the usual manner.

2 Recording data with keyboard entries (eg titrations, Boyle's Law)

The first step is to click on **Experiments** and select **Show Sensors**. This displays a Sensors(Lab Pro) window, listing icons of all the available sensors. Drag the appropriate sensor to the appropriate channel (CH1 – CH4 for the analogue sensors on the left of the window, DIG/SONIC for the digital sensors on the right of the window). After attaching the sensor(s), click on the Close button. This results in a data table and graph appropriate to the sensor being displayed. The data table is headed **Latest**, with Time as the X variable and the property measured by the sensor as the Y variable.

The second step is to click on **Experiments** to set up the data collection. From the drop-down menu, select the **Data Collection** option. This displays a **Data Collection** window with two tabs, labelled **Collection** and **Triggering**, respectively. Change the default Mode entry (Time Based) to **Events with Entry** and make the appropriate entries in the **Column Name**, **Short Name** and **Units** boxes and click on the **Done** button. This results in a data table and graph being displayed, with the keyboard entries displayed as the X variable.

3 Recording Data from two sensors

The first step is to click on **Experiments** and select **Show Sensors**. This displays a Sensors(Lab Pro) window, listing icons of all the available sensors. Drag the appropriate sensors to the appropriate channels (CH1 – CH4 for the analogue sensors on the left of the window, DIG/SONIC for the digital sensors on the right of the window). After attaching the sensors, click on the Close button. This results in a three column data table data table and two graphs, appropriate to the sensors being displayed. The data table is headed **Latest**, with Time as the X variable and the properties measured by the sensors as the Y1 and Y2 variables.

The second step is to click on **Experiments** to set up the data collection. From the drop-down menu, select the **Data Collection** option. This displays a **Data Collection** window with two tabs, labelled **Collection** and **Triggering**, respectively. The default setting is to collect data in a continuous stream (**Time Based** mode). The duration of the data collection is set by entering the appropriate number and time unit in to the **Length** boxes (time units available are milliseconds, seconds, minutes and hours). The sampling rate is set either by moving a slider bar until the appropriate samples/second or seconds/sample is shown or else by entering the appropriate number in the box. (For oversampling, see later). Click the **Done** button to return to the screen.

At this point, there will be a separate display for each sensor.

To display data from both sensors on the same graph

Click first on one of the two graphs and then on *Options*. Select *Graph Options* – this leads to a *Graph Options* window with two tabs, labelled *Graph Options* and *Axes Options*. The default (*Graph options*) allows a title to be given to the graph, error bars to be shown and connecting lines to be drawn between data points. Clicking on the *Axes Options* tab displays the *Axes Options* window. A box labelled Y Axis Columns displays check boxes for time and the two sensors, only one of which will contain a check mark.

To display data from both sensors on the same graph, place a check mark in the appropriate empty box and then click the *Done* button. Right Click on the graph which is not required and select *Delete*. At this point there will be one graph with the Y axis labelled with both sensor outputs, occupying approximately half the screen. The graph can be re-sized by clicking and dragging on any of the small black squares on the graph border.

To plot one sensor output against the other

Proceed as above to the point where the *Axes Options* tab displays the *Axes Options* window. Change the X-Axis label by clicking on the downward pointing arrow in the X Column box. Click on *Options* and select *Graph options* from the drop-down menu and select the appropriate sensor measurement. Click on *Done* to complete the setting up.

Advanced Features

Oversampling

The LoggerPro interface is able to collect data over the range of 50,000 second⁻¹ to one sample in 4.4 hours. Over part of this range, it is possible, by oversampling, to collect a number of data samples and average them. The purpose of this is to reduce the amount of noise in the signal from the sensor.

Eg if a count rate of 1 sample every 3 seconds is chosen, 300 samples are taken during that 3 second period and averaged. The number of samples being averaged depends on the sample period – in fast counting, only a few samples will be averaged: at slow count rates, the number of samples collected and averaged is much greater. The number of samples being averaged is indicated in brackets.

Curve Fitting

Click on the Curve Fit icon or select Curve Fit from the Analyse menu to display the Curve Fit window. This displays a small graph and a table of General Equation options. Beneath the table is a box which allows the user to define a custom curve fit.

The option needs only the right hand side of the equation defining the customised curve fit –

eg in a Boyle's law experiment, where the data columns would be labelled time, Pressure and Volume, to define Inverse Volume the entry would be $1/\text{Volume}$

When an equation has been chosen, click on the Try Fit button. The value of each coefficient is then displayed in the Coefficients table and the curve fit is displayed in the reduced graph. The number of significant figures displayed in the coefficients can be set from the File menu (*Preferences, Applications Preferences, Default Precision*)

Multiple Graphs

When a calculated column has been produced, its data can be displayed on a separate graph, inset in the main graph. To do this, simply click on *Insert* and select *Graph* – drag the graph to a clear area of the screen and resize as necessary.

1st and 2nd Derivatives

Where it is useful to plot the first or second derivative of a data set, click first on *Data* on the title bar and select *New Calculated Column*. Insert the appropriate entries in the boxes in the pop-up window and then click on the *Functions* button. Select *Calculus* from the options and then *derivative* (for the 1st derivative) or *secondDerivative* (for the 2nd derivative). This results in a new column being added to the data table for each derivative chosen. To show the derivative plot, select *Insert* from the title bar and *Graph* from the drop-down menu. This will display the derivative plot on a separate graph, which may be resized and dragged to a clear area of the screen as appropriate.

All this may seem rather intimidating but, in practice, you will find that data logging with the Vernier interface and sensors simply involves running your experiment **as you would normally do**, but with a sensor to collect that data and your computer to store and present it.

Determination of the Orders of Reaction (Phenolphthalein/OH⁻ Reaction)

Introduction:-

Phenolphthalein is an indicator which shows a strong pink colour in alkaline solution. In moderately concentrated solutions, the pink colour fades as a result of a reaction between the red anion and hydroxide ions to form a colourless complex.

The rate of this reaction depends, in some way, upon the concentration of the hydroxide ions and the concentration of the phenolphthalein:-

$$\text{Rate of reaction} = k[\text{Ph}^-]^x[\text{OH}^-]^y$$

where x and y are the orders of reaction with respect to phenolphthalein and hydroxide ions respectively.

Procedure:-

Plug in the Colorimeter into the Vernier interface and connect to the computer by way of the USB slot. Click on File on the title bar and select "Fading" from the **Experiment** folder to set up the computer to record absorption at a wavelength of 468 nm (blue). Zero the instrument by filling the cell with distilled water and pressing the **Calibrate** button on the colorimeter. Click on **Data** on the title bar and select **Data Collection**. Set the interface to collect data for 4 minutes, at 5 second intervals.

Place 3.0 cm³ of sodium hydroxide solution (1.0 mol dm⁻³) in a clean dry boiling tube. Add 7.0 cm³ of distilled water. Invert the tube to mix the contents and transfer 3 cm³ solution to the colorimeter cell. Add 3 drops of 0.1% phenolphthalein solution and invert the cell to mix the reactants quickly.

Press the **Collect** button to collect data until the absorbance decreases to less than 25% of its initial value. End the data logging by pressing the **Stop** button. From the **File** menu, select **Export** to save your data to your personal data disc, using the file-name P1. Discard the solution and rinse the cell with distilled water.

Prepare the following reaction mixtures, but not until you are ready to measure the rate of reaction of each mixture:-

Run No.	2	3
NaOH/cm ³	6.0	9.0
Water/cm ³	4.0	1.0

Record the absorbance of each mixture until the value decreases to less than 25% of the initial value. Export your data from each run before proceeding to the next mixture, saving your data for the second run in a file called P2 and data for the third run in a file called P3.

Results:-

Click on **Analyse** in the title bar and select **Curve Fit**. Scroll down the list of options in the General Equation window. Select the **natural Exponent** fit and click on the **Try Fit** button.

Repeat this for each of the data sets.

Set the printer to Landscape orientation and print the graph showing the three absorbance vs time graphs.

Reference:- Kinetics of Fading, Nicholson J, J Chem Ed., **66** 725 - 726 (1989)

This article gives the structures of the reactants and products, together with the mechanism and a value for the rate constant.

For demonstration purposes, sodium hydroxide concentrations of 1 mol dm^{-3} and 2 mol dm^{-3} , and a time interval of 3 seconds may be used for faster results.

Determination of the Indicator Constant of Bromothymol Blue

INTRODUCTION:-

Acid-base indicators generally behave like weak acids, HIn , which ionise -



In strongly acid solutions, bromothymol blue exists in the yellow, molecular form HIn. In strongly alkaline solutions it occurs as the bright blue ionised form, the colour being due to the In^- anion. In solutions between these two extremes, the indicator exists as a mixture of the yellow and blue forms giving solutions which range in colour from slightly greenish yellow to slightly greenish blue through a continuous range of shades of green.

The dissociation constant (K) for bromothymol blue (HIn) can be written as –

$$K = \frac{[\text{H}^+][\text{In}^-]}{[\text{HIn}]}$$

Taking logs (to base 10) of both sides leads to –

$$\log K = \log[\text{H}^+] + \log \frac{[\text{In}^-]}{[\text{HIn}]}$$

Moving log K to the right hand-side and log $[\text{H}^+]$ to the left hand-side (and remembering to change signs as well!) leads to –

$$-\log[\text{H}^+] = -\log K + \log \frac{[\text{In}^-]}{[\text{HIn}]}$$

In this experiment, the ratio of ionised to molecular forms of the indicator is calculated from measurements of absorbance made at 635 nm (red). At this wavelength, the absorbance of the blue (ionised) form is at a maximum (A_{In}) and the absorbance of the yellow (molecular) form is very small (A_{HIn}).

A set of buffer solutions is prepared which spans the 6.2 - 8.2 pH range and all of which contain a constant concentration of bromothymol blue. In these solutions, the fraction of the indicator which is in the blue (ionised) form increases as the pH increases.

For most of the solutions, the different shades of green should be quite easily apparent but in the highest pH solutions this may not be the case, as the sensitivity of the eye varies in different individuals. This limits the range of pH over which the colour is seen to change - for most indicators this pH range is approximately two units.

PROCEDURE:-

Three stock solutions are provided – 0.05% bromothymol-blue indicator solution, $0.100 \text{ mol dm}^{-3}$ disodium hydrogen phosphate (DSHP) and a solution containing $0.100 \text{ mol dm}^{-3}$ potassium dihydrogen phosphate (PDHP) and $0.200 \text{ mol dm}^{-3}$ potassium chloride.

Preparation of Buffer Solutions

Label nine 25 cm^3 graduated flasks from 0 to 8 and then prepare the following mixtures, taking particular care in measuring the bromothymol blue solution:-

Solution No.	Bromothymol Blue/ cm^3	DSHP/ cm^3	PDHP/ cm^3
--------------	---------------------------------	---------------------	---------------------

0	1.0	0.0	10.0
1	1.0	1.0	9.0
2	1.0	2.0	8.0
3	1.0	3.0	7.0
4	1.0	4.0	6.0
5	1.0	5.0	5.0
6	1.0	6.0	4.0
7	1.0	7.0	3.0
8	1.0	10.0	0.0

Make up the contents of each flask to 25 cm³ with distilled/deionised water and shake thoroughly before making any measurements.

Data Logging – Part 1

Insert the colorimeter into the Vernier interface and connect the interface to the computer USB port. Fill the colorimeter cell with distilled water, place it in the colorimeter and calibrate the sensor by pressing the Calibrate button on the colorimeter. Select BTBlue_1 from the Experiments folder to download the data logging settings for this part of the experiment.

Fill a clean, dry 1 cm cell with solution 0, place it in the colorimeter and click on the COLLECT button. When the reading is steady, click on the KEEP button and enter 0 in the pop-up window.

Return the solution to its flask and then rinse the cell twice with solution 1. Fill the cell with solution 1 and record the absorbance and the number of the flask as before.

Proceed in this way to measure the absorbance of the remaining solutions, end the data recording by pressing STOP and export the data to a file called BT1.

Data Logging – Part 2

Connect the pH sensor to the interface and calibrate, if necessary, with buffer solutions of pH = 4 and either pH = 7 or pH = 9. (This will normally be done for you.).

Select BTBLue_2 from the Experiments folder to set up the data logging procedure. Place the pH sensor in solution 0 and click on the Collect button. When the reading is steady, record the pH of the solution and enter the number of the flask in the pop-up window.

Rinse the sensor with a small portion of the next solution and then record the pH of the main solution by clicking on the Keep button and entering the number of the solution. Click on the STOP button after you have measured the

last pH. Export the data to a file called BT2. Rinse the pH sensor with distilled water and return it to its storage bottle..

RESULTS:-

The absorbance of solution 0 gives the value of A_{HIn} and the absorbance of solution 8 gives the value of A_{In} . Solutions 1 - 7 contain varying fractions (α) of the indicator in the ionised form and corresponding fractions ($1 - \alpha$) in the molecular form.

The absorbance (A) of these solutions is therefore given by -

$$\begin{aligned} A &= \alpha A_{In} + (1 - \alpha) A_{HIn} \\ &= \alpha A_{In} + A_{HIn} - \alpha A_{HIn} \\ &= A - A_{HIn} = \alpha A_{In} - \alpha A_{HIn} \\ &= \alpha (A_{In} - A_{HIn}) \\ \alpha &= \frac{A - A_{HIn}}{A_{In} - A_{HIn}} \end{aligned}$$

Since this gives us the fraction (α) of the indicator in the ionised form, the ratio of $\alpha/(1 - \alpha)$ can easily be shown to be :-

$$\frac{\alpha}{1 - \alpha} = \frac{A - A_{HIn}}{A_{In} - A_{HIn}}$$

Since all of the solutions contain the same total concentration of bromothymol blue, this ratio can be substituted to give as the working equation -

$$pH = pK + \log \frac{A - A_{HIn}}{A_{HIn} - A_{In}}$$

which indicates that if the pH is plotted against the log of this ratio, the resulting graph should be linear with a slope of unity and an intercept which gives the value of pK_a . The dissociation constant of the indicator can then be calculated from its pK_a value.

These calculations are contained in the BTBlue spreadsheet.

NOTE:-

Potassium chloride is included in the potassium dihydrogen phosphate solution to ensure that a property of the mixtures called the ionic strength remains constant. The experiment would still work without it, but the graph would be slightly less linear.

PROBLEMS FOR DISCUSSION:-

1. Relate the range of solution colours which you can distinguish to the pH values. To what extent could you use the colour of a solution to estimate its

pH value? Does the kind of lighting (i.e. daylight, tungsten, fluorescent) have any effect on your ability to recognise /match colours?

2. Is the working range of bromothymol blue approximately 2 pH units? How is the working range related to the pK value of the indicator?
3. Compare the pK_a value with that given in the Rubber Handbook or any similar Data Book. Is there good agreement or a significant difference?
4. If your graph is not linear, can you account for the most probable source of errors?
5. Litmus contains an indicator with a pK_a very similar to that of bromothymol blue but its colour change is from red in acid solution to blue in alkaline solution. Which of the two indicators is likely to be the better indicator if you have to estimate the pH of a solution from its colour?
6. The pK values of some acids, bases and indicators are given in the following table:-

Acid/Base	pK	Indicator	pK
Ammonia	9.24	Bromocresol Green	4.7
Ethanoic Acid	4.74	Methyl Orange	3.7
Hydrazine	7.94	Phenol Red	7.9
Methanoic Acid	3.75	Thymolphthalein	9.2

If you were to plan a similar experiment with these indicators, match the indicator to the appropriate acid.

Reference:-

A Simplified Method of Finding K_a of an Acid-Base Indicator, G S Patterson, J Chem Ed, **76**, 395 –7, (1999)

Investigation of the Iron(III) - Hydroxybenzoate Complex (Job's Method)

Introduction:-

In the first part of this experiment, the formula of a complex ion is determined by Job's method of continuous variation. This involves the preparation of two solutions of exactly equal concentration. A series of mixtures is made up ranging from pure iron(III) to pure hydroxybenzoic (salicylic) acid in which the total concentration of solutes is constant.

At either end of the range, there is a large excess of one reactant and the concentration of the complex is determined by the concentration of the reactant which is not in excess. Towards the middle, the excess of one reactant over the other gets smaller and at some point, the two reactants are present in the ratio which gives the maximum amount of complex. When the equilibrium constant is very large, a sharp inverted V type graph is obtained. When the equilibrium constant is not so large, the graph has a more rounded appearance.

The iron(III) - hydroxybenzoate complex is purple in colour and so can be determined colorimetrically. The absorbance of the solution plotted against the composition of the mixture gives a graph with the maximum occurring at the ratio of iron(III) to hydroxybenzoate in the complex.

In the second part of the experiment, the concentration of the complex is estimated in three of the solutions. From this, it is then possible to determine the concentrations of the remaining iron(III) and hydroxybenzoic (salicylic) acid and then the equilibrium constant. Equilibrium constants are almost meaningless unless they are reported with the temperature at which they were measured. It is important to record the temperature of the solutions since this is required in the final calculation.

Procedure:-

1. Determining the formula:-

Weigh exactly 0.260 - 0.270 g of ammonium iron(III) sulphate into a beaker and dissolve it in warm dilute hydrochloric acid (0.1 mol dm^{-3} , 20 cm^3). Transfer the solution to a 1 dm^3 graduated flask and rinse the beaker several times with distilled/deionised water and add these volumes of water to the flask. Finally, make up to the mark with distilled/deionised water. Shake thoroughly to ensure complete mixing. This solution should be colourless - if it is any shade of brown, hydrolysis has occurred and a fresher sample of ammonium iron(III) sulphate is required.

Weigh exactly 0.135 - 1.40 g of hydroxybenzoic acid into a beaker and dissolve it in warm distilled/deionised water (50 cm^3). Add dilute hydrochloric acid (0.1 mol dm^{-3} , 20 cm^3) and transfer the solution to a 1 dm^3 graduated flask. Rinse the beaker several times with distilled/deionised water and add these volumes of water to the flask. Finally make up the contents of the flask to the mark with distilled/deionised water. Shake thoroughly to ensure a uniform solution.

Label clean dry boiling tubes 0 - 10 and make up the following mixtures, dispensing the solutions from a burette or graduated pipette:-

Tube No.	0	1	2	3	4	5	6	7	8	9	10
$\text{Fe}^{3+}/\text{cm}^3$	0.0	1.0	2.0	3.0	4.0	5.0	6.0	7.0	8.0	9.0	10.0
HBA/cm^3	10.0	9.0	8.0	7.0	6.0	5.0	4.0	3.0	2.0	1.0	0.0

(HBA = hydroxybenzoic acid)

Place a thermometer in tube 10 and record the temperature.

Insert the colorimeter into the Vernier interface and connect the interface to the USB port of the computer. Select the green LED so that the absorbance can be measured at 565 nm. Select the JOB file from the Experiments folder to download the computer settings for the experiment.

Rinse the cell with solution 0 and then refill the cell. Start the data logging by clicking on the COLLECT button. When the absorbance reading is steady, lick on KEEP and enter the tube number, from the keyboard in the pop-up window.

Discard the solution, rinse the cell twice with the next solution and refill it. Record the absorbance and the tube number of this solution as before. Proceed in this way through the remainder of the solutions and, finally, end the data logging by clicking on the STOP button. Before discarding the last solution, record its temperature. Follow the on-screen instructions for treating the data and then export the data to a file called Job1.

2. Determining the equilibrium constant:-

Pour approximately 50 cm³ of the ammonium iron(III) sulphate solution into a clean dry beaker and add approximately 0.5 g of hydroxybenzoic acid. Stir the mixture vigorously and then filter the solution through a fast filter paper. Collect the first 5 cm³ of filtrate in a test tube and discard it. Collect the remaining filtrate in a clean dry flask or beaker.

In four 10 cm³ graduated flasks prepare the following dilutions of the complex solution:-

Flask no.	1	2	3	4
Complex/cm ³	2.0	4.0	6.0	8.0
Water/cm ³	8.0	6.0	4.0	2.0

Shake thoroughly to ensure good mixing. Record the absorbance of distilled/deionised water (enter flask number as 0) and the absorbance of each of the above solutions, together with the absorbance of the filtrate in the beaker (enter flask number as 5). Follow the on-screen instructions for constructing a calibration graph and then export the data to a file called Job2.

RESULTS:-

1. Determining the formula:-

Open the **Job** spreadsheet and check that your data have been imported. Enter the masses of ammonium iron(iii) sulphate and hydroxybenzoic acid used and complete the graph by drawing the best straight lines through the linear portions of the graph.

Draw a line down from where the lines cross to the axis and calculate the concentration of iron(III) and hydroxybenzoic acid in that particular mixture. The ratio of these concentrations gives the ratio of iron(III) to hydroxybenzoic acid in the complex.

2. Determining the equilibrium constant:-

The addition of excess hydroxybenzoic acid converts all the ammonium iron(III) sulphate in the original solution into the complex salt. The concentration of the complex ion in this solution is therefore $0.0010 \text{ mol dm}^{-3}$. The concentration of complex in each of the diluted solutions is calculated and a calibration graph of absorbance vs complex concentration is produced.

Use this calibration graph to calculate the concentration of the complex in solutions 4,5 and 6. This allows the concentration of the remaining iron(III) and hydroxybenzoate to be calculated and from this, the equilibrium constant can be calculated.

Tube No.	4	5	6
Initial $[\text{Fe}^{3+}]/\text{mol dm}^{-3}$			
Initial $[\text{HBA}]/\text{mol dm}^{-3}$			
$[\text{Complex}]/\text{mol dm}^{-3}$			
Remaining $[\text{Fe}^{3+}]/\text{mol dm}^{-3}$			
Remaining $[\text{HBA}].\text{mol dm}^{-3}$			
Equilibrium Constant/ $\text{dm}^3 \text{ mol}^{-1}$			

Average value of equilibrium constant:- $\text{dm}^3 \text{ mol}^{-1}$ at K

$$\begin{aligned} \Delta G^0 &= -RT \log_e K \\ &= \text{..... J mol}^{-1} \\ &= \text{.....kJ mol}^{-1} \end{aligned}$$

Determination of the Enthalpy of Neutralisation

Introduction:-

Neutralisation is the reaction which occurs when a base is added to an acid to give a salt and water as the only products. In this experiment, the change in temperature when this kind of reaction occurs is measured by a data-logging system. The graph which is produced can be used to estimate the highest temperature reached by the reacting mixture.

The data can be transferred to a spreadsheet, together with other data - such as the volumes of acid and base used, together with their concentrations - to calculate how much heat (the enthalpy of neutralisation) would have been

produced if 1 mole of the base had neutralised several different acids.

Procedure:-

Insert the temperature sensor into the Vernier interface and connect the interface to the computer USB port. Place the sensor in a beaker of distilled water until it is required. Select the Enthalpy of Reaction file from the Experiments folder to download the appropriate settings for this experiment. Click on Experiment on the title bar and select Data Collection to check that data will be collected at 2 second intervals.

Measure as accurately as possible, using a measuring cylinder, 25 cm³ of 2.0 mol dm⁻³ sodium hydroxide solution (**Caution - sodium hydroxide solution is corrosive!**) into a polystyrene beaker. Place the temperature sensor in the solution.

Measure 26 cm³ of 2.0 mol dm⁻³ hydrochloric acid into a clean dry beaker (**Caution - hydrochloric acid is corrosive!**).

Click on the COLLECT icon to begin recording the temperature of the sodium hydroxide solution. After 30 seconds, quickly add the hydrochloric acid and stir gently with the temperature sensor. Continue to record the temperature for a further two minutes.

Set the printer to Landscape orientation and print the graph straightaway, otherwise export the data to a file called N1 on your floppy disc. Clear the screen ready to display the next set of data.

Discard the solution and rinse the beaker with distilled water. Dry the beaker with a paper tissue and then repeat the experiment but using 2.0 mol dm⁻³ nitric acid instead of hydrochloric acid (calling the file N2 if the data are to be saved on disc).

If time permits, repeat the procedure with -

a) 2.0 mol dm⁻³ ethanoic acid (file name N3)

b) 1.0 mol dm⁻³ sulphuric acid (file name N4)

Print the temperature vs time graphs immediately after each neutralisation.

Data Processing:-

On each of the neutralisation graphs, continue the cooling curve back to the time when the acid was added to the sodium hydroxide solution. Estimate the rise in temperature of the solution which would have occurred if there had been no loss of heat to the surroundings.

Open the **Neutralisation** spreadsheet and enter the volumes of acid and base which were used, together with the concentration of the sodium hydroxide solution.

Print the completed spreadsheet and then use the information to answer the following questions:-

- 1 Is the enthalpy of reaction roughly the same for each combination of acid and base?
- 2 What is the point of drawing the temperature vs time graphs?
- 3 What assumptions have been made in calculating the enthalpies of neutralisation?

Determination of a Solubility Curve

Introduction:-

Solubility is defined as the mass of a solute which will saturate 100 g of solvent at a specified temperature. Since the dissolving process involves breaking down the solid structure of the solute, it is an endothermic reaction for most solutes. The positive ions, however, tend to attract the lone pair electrons of the water molecules in a process called hydration. To a lesser extent, the negative ions tend to attract the partially charged hydrogen atoms of the water molecules. Although these hydration processes are exothermic, they are not usually large enough to make the complete solution process exothermic. Solubility, therefore, usually increases with increasing temperature.

The LoggerPro software has been set up to calculate the solubility of the solute as the data are collected. The equation which it will use is given in the Results section and the software will prompt you to enter the volume values from the computer keyboard

Procedure:-

Double click the **Solubility** icon to set up the computer and temperature sensor to record and display data. Plug the temperature sensor into the Vernier interface and connect the interface to the computer USB port.

Weigh a clean dry boiling tube, add 16 - 17 g of potassium nitrate and weigh again. Record the mass (M) of potassium nitrate. Follow the on-screen instructions which will allow the solubility to be plotted directly.

Add 10.0 cm³ of distilled water from a burette and insert the temperature sensor. Heat the mixture, stirring gently with the sensor until the potassium nitrate has dissolved completely.

Press the **Collect** button on the computer screen and allow the hot solution to cool, stirring continuously with the temperature probe. The temperature value is displayed on the screen. When the first crystals appear, click on the **Keep** button to record the temperature at which this occurs and, from the keyboard, enter 10 in the pop-up window on the screen.

Add 1.0 cm³ of water from the burette and heat the mixture until the potassium nitrate has dissolved completely. Allow the solution to cool, stirring continuously with the temperature probe. Record the temperature at which the first crystals appear and enter 11 from the keyboard.

Continue in this way until a total of 8 cm³ of distilled water has been added.

Set the printer to Landscape orientation and print the graph. Finally, export the solubility and temperature data to a file called S1 on your personal data disc.

Results:-

The LoggerPro software uses the temperature and volume data to produce a solubility curve.

The data processing involves the following steps:-

$$\begin{aligned} \text{Mass of boiling tube + solute} &= (M + m) \text{ g} \\ \text{Mass of empty boiling tube} &= m \text{ g} \\ \text{Mass of solute} &= M \text{ g} \\ \text{Initial volume of water} &= 10 \text{ cm}^3 \\ \text{Volume of water added} &= v \text{ cm}^3 \\ \\ \text{Solubility of potassium nitrate} &= 100 M / (10 + v) \text{ g}/100 \text{ cm}^3 \end{aligned}$$

Further work:-

Other suitable solutes include 2-hydroxybenzoic acid (salicylic acid), lead chloride, lead bromide and lead iodide.

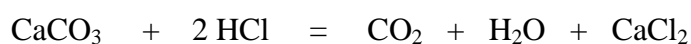
Advanced pupils can create a spreadsheet which will plot \ln Solubility vs $1/T$ (Kelvin) to obtain the enthalpy of solution for a particular solute.

Reference:-

Solubility of Lead Halides, D M Lichty, JACS, **25**, 471 (1903)

To Investigate the Factors Affecting the Rate of the Marble / Hydrochloric Acid Reaction**Introduction:-**

The rate of a chemical reaction depends upon a number of factors. This experiment examines the effect of changing the concentration of the acid. When marble chips are dropped into hydrochloric acid, carbon dioxide gas is formed and a solution of calcium chloride is left behind:-



Although the rate of the reaction could be measured by measuring the volume of gas produced as the marble dissolves, it is easier to measure the pressure developed by the gas if it is not allowed to escape from flask. A pressure

sensor is attached to a small filter flask or boiling tube to measure the pressure at short time intervals set up by the computer program.

Procedure:-

Connect the pressure sensor to the filter flask or boiling tube with a side arm by a short length of pressure tubing. If a boiling tube with side arm is not available, the pressure tubing should be inserted into a one-holed rubber bung.

Insert the pressure sensor into the Vernier interface and connect the interface to the USB computer port. Select **marble** from the Experiments folder to download the data logging settings for this experiment.

The experiment is most easily done by using a two person team, one member being responsible for the data-logging and the other being responsible for the chemical operations.

Team member A

Place 10 cm³ of distilled water and 10 cm³ of 2 mol dm⁻³ hydrochloric acid in the boiling tube and measure the temperature of the resulting mixture. Take one large (2 - 3 g) marble chip and drop it into the acid when team member B is ready to start data logging.

When team member B is ready, drop the marble chip into the acid. Wait for approximately 15 seconds for the carbon dioxide to saturate the solution and then insert the bung firmly and hold it in position until sufficient data have been collected. Swirl the tube gently to ensure that the gas is liberated steadily.

When team member B gives a signal that enough data have been collected, remove the bung and pour away the solution. Rinse the marble chip, place it on a filter paper and rinse the flask.

Repeat the experiment with the same marble chip, but using 15 cm³ of 2 mol dm⁻³ hydrochloric acid and 5 cm³ of water.

Repeat the experiment but using 20 cm³ of 2 mol dm⁻³ hydrochloric acid. Finally wash the flask and return it to its storage place.

Team member B

Data logging begins when you click on the START button and ends when you click the STOP button. When you are ready to start, tell team member A to add the marble to the acid. When the bung goes into the flask, wait for 10 - 15 seconds before starting the data logging. (This is to allow the solution to become saturated with carbon dioxide.)

Click on the START button and then stop the data logging when the pressure reaches 130 kPa by clicking on the STOP button. Tell team member A that

you have finished. (It is important to stop data logging before the bung is removed from the filter flask/boiling tube.)

Repeat this procedure for each of the different acid concentrations. Finally, print two copies of the graph, making sure that the printer is set to print in Landscape format.

Data Processing and Interpretation:-

Either draw the best straight lines through the sets of data and measure the slope of each line or else use the Fit function, and then answer the following questions:-

Questions to consider:-

- 1 By the end of the experiment, enough carbon dioxide has been produced to give an increase in pressure of approximately 30 kPa in a flask with a volume of $250 \times 10^{-6} \text{ m}^3$ at a temperature close to 298 K. If the gas constant $R = 8.314 \text{ J K}^{-1} \text{ mol}^{-1}$, how many moles of carbon dioxide have been formed ?
- 2 How many moles of calcium carbonate reacted?
- 3 If the mass of 1 mole of calcium carbonate is 100 g, has the marble chip suffered a large change in mass ?
- 4 How many moles of hydrochloric acid were used up in this reaction ?
- 5 Did the concentration of hydrochloric acid change very much during the reaction ?
- 6 Why was the same marble chip used on each part of the experiment ?
- 7 Is there any connection between the three initial rates of reaction and the three hydrochloric acid concentrations ?
- 8 If nitric acid of the same concentrations were to be used instead of hydrochloric acid, would you expect the reaction to be slower, faster or about the same? Give one reason for your answer.
- 9 If ethanoic acid of the same concentrations were to be used instead of hydrochloric acid, would you expect the reaction to be slower, faster or about the same ? Give one reason for your answer.
- 10 How could this experiment be altered to show the effect of temperature on the rate of reaction?

pH Changes in Acid/Base Titrations

Introduction:-

In pH titrations, an electrode, made of a special kind of glass, responds to the changing concentration of hydrogen ions. The charge on the electrode (ie the electrode potential) is compared with the potential of a standard (constant) electrode (usually a saturated calomel electrode or a silver/silver chloride electrode contained inside the glass electrode). All combinations of strong/weak acid against strong/weak base can be monitored with such an electrode, except the weak acid/weak base titration.

PROCEDURE:-

Plug the pH sensor into the Vernier interface and then connect to the USB port on the computer. Select the **Titration** file from the Experiments folder to download the data logging settings for this experiment. Calibrate the pH sensor using two buffer solutions (pH = 4 and pH = 9). When not in use, the

pH sensor should be placed in a beaker of distilled water or returned to its storage container - it should not be allowed to dry out.

Place, by means of a pipette, 25.0 cm³ of standard hydrochloric acid solution in a clean 250 cm³ beaker and add approximately 50 cm³ of distilled water. Swirl the contents to ensure good mixing. Place the pH probe in the solution.

Fill a burette with the sodium hydroxide solution of “unknown” concentration, making sure that there is no air bubble left under the tap and that the initial reading is 0.0 cm³. Click on the COLLECT button to display the pH value. When the reading is steady, click on the KEEP button and enter 0 in the pop-up window.

Run in 5 cm³ of the solution and swirl the contents or use the pH sensor as a stirring rod. Record the pH of the solution and enter the volume of sodium hydroxide, from the key-board in the pop-up window. Repeat this procedure until a total of 20 cm³ of solution has been added. From this point, add the sodium hydroxide in 1 cm³ portions until a total of 30 cm³ has been added.

From this rough titration, estimate where the end-point of the titration occurs, then clear the screen and discard the data. (Click on Data and select the Delete Data option.) Discard the solution and rinse the flask.

Repeat the titration but as the end-point approaches, add the sodium hydroxide solution in much smaller portions (0.1 cm³), recording the pH after each addition. After the end-point, continue to add sodium hydroxide solution in 1 cm³ portions until the total volume added reaches approximately 30 cm³. Export the data with the file-name **pH1**.

Select **New Calculated Column** from the Data drop-down menu and then enter 1st Derivative in both the Name and Short Name boxes in the pop-up window. Enter mL in the Units box and then click on the Functions button. Select **Calculus** and **derivative** from the list of options that then appears. In the **Equation** box, enter “pH” (with the quotes) inside the pair of bracket and click Done.

Click on **Insert** on the title bar and select graph. Delete the data table, rescale the graphs and arrange them so that they fill the screen. Check that the printer is in Landscape orientation and print the graph. Clear the screen by deleting each graph in turn.

After the titration has been completed, empty the contents of the flask and rinse the flask with distilled/de-ionised water. Return the pH sensor to its beaker of distilled water.

Repeat the experiment, but replacing the hydrochloric acid solution with a standard ethanoic acid solution. Export the data using the file-name **pH2**.

Fill a clean burette with an ammonium hydroxide solution of “unknown” concentration, taking the same precautions as before. Place 25 cm³ of standard

hydrochloric acid solution in a clean titration flask and titrate the solution as described above.

After the titration has been completed, empty the flask and rinse it with distilled/de-ionised water. Export the data using the file name **pH3**.

RESULTS:-

Identify the end points in each titration and then calculate the concentration of each acid and of the ammonia solution.

Use the **Titration** spreadsheet to display your titration data and to show an alternative method of finding the end-point.

Questions for Discussion:-

1. How can you tell where the end-point occurs in each titration ?
2. Calculate the concentration of each of the “unknown” solutions.
3. What differences are there between:-
 - a) the sodium hydroxide/hydrochloric acid titration and the sodium hydroxide/ethanoic acid titration ?
 - b) the sodium hydroxide/hydrochloric acid titration and the ammonium hydroxide/hydrochloric acid titration ?
4. What property do you expect to be shown by a solution which contains either a weak acid and one of its salts, or a weak base and one of its salts ?
5. The approximate pH of a solution containing a weak **acid** and one of its salts is given by the following equation:-

$$pH = pK_a + \log \frac{[Salt]}{[Acid]}$$

What is especially important about the pH when the titration is half-complete?

Determination of pK_a of a Weak Acid

Introduction:-

When a solution of a weak acid is titrated with a strong base, the pH of the solution rises only very slowly until quite close to the equivalence point. This is because the solution contains both the weak acid, yet to be titrated, and one of its salts, formed during the titration.

These are the components of a buffer solution and the most important feature of a buffer solution is that its pH changes only very slightly when a moderate amount of acid or base is added to it.

The pH of the buffer solution depends upon the pK_a of the acid and the ratio of the salt and acid concentrations. If the weak acid is represented by HX, then its dissociation can be represented by -



The equilibrium constant for this reaction is written as -

$$K_a = \frac{[H^+][X^-]}{[HX]}$$

This can be split to give -

$$K_a = [H^+] \times \frac{[X^-]}{[HX]}$$

Taking logs of both sides leads to -

$$\log K_a = \log[H^+] + \log \frac{[X^-]}{[HX]}$$

Moving $\log K_a$ to the right hand side of the equation and $\log [H^+]$ to the left hand side, leads to -

$$-\log[H^+] = -\log K_a + \log \frac{[X^-]}{[HX]}$$

or

$$pH = pK_a + \log \frac{[X^-]}{[HX]}$$

By measuring the pH at several points before the equivalence point and plotting the pH against the log of the ratio of salt and acid concentrations, the pK_a of the acid is given by the value of the intercept.

(It is important to remember that the salt of a weak acid undergoes hydrolysis and this will affect the concentration of both the anion and the undissociated weak acid.)

Procedure:-

Insert the pH sensor into the Vernier interface and connect the interface to the USB port. Standardise the sensor, if necessary, with buffers of pH 4 and pH 9. Place the sensor into a beaker of distilled water until required.

Fill the burette with a standardised solution of sodium hydroxide (0.10 mol dm^{-3}) and check that there is no air bubble under the tap and that the tap does not leak. Place 25 cm^3 of standardised weak acid solution, by pipette, into a clean 250 cm^3 beaker and add approximately 80 cm^3 of distilled water. Swirl the contents of the beaker to ensure the solution is well mixed.

Place the pH probe in the solution and click on the COLLECT button. Add 4 cm^3 of sodium hydroxide solution and swirl the solution. When the pH reading is steady, press the KEEP button and enter the volume of sodium hydroxide (from the keyboard).

Continue, in this way, to record the pH of the solution after the addition of 4 cm^3 portions of sodium hydroxide solution until a total of 20 cm^3 has been added. End the data logging by clicking on the STOP button. Export the data to a file called pK1 on your floppy disc.

Repeat the partial titration after clearing all data from the screen and export the second set of data to a file called pK2 on your floppy disc.

Data Analysis:-

Open the pK_a spreadsheet and check that your data have been imported. Compare the values of pK_a obtained in this experiment with the literature value reported in your data book.

Conductivity Changes in Acid/Base Titrations

Introduction:-

Conductimetric titrations depend upon the following observations:-

- a) hydrogen ions travel faster in aqueous solution than any other ions
- b) hydroxide ions travel at approximately half the speed of hydrogen ions
- c) most of the other kinds of ions travel at approximately half the speed of hydroxide ions

During a conductimetric titration, a small alternating current is passed between two platinum electrodes placed in the reaction vessel. The current is small, so as not to affect the chemical composition of the solution which is being titrated.

Alternating current is used so that the electrodes do not get coated with the products of electrolysis (the alternating current causes the electrodes to change rapidly from cathode to anode and vice versa, thus cancelling out any electrolysis changes).

These titrations do not require the presence of an indicator to signal the end-point and therefore can be used in the titration of coloured solutions such as fruit juices. Conductimetric titrations do not require the end-point to be approached carefully as the end-point is determined graphically.

In the case of a strong acid titrated with a strong base, the conductivity is initially large, since the strong acid is completely ionised and there is a large concentration of hydrogen ions. As the strong base is added, the hydroxide ions react with hydrogen ions to produce water and the conductivity decreases.

After the end-point, hydroxide ions accumulate in the solution and the conductivity increases. The graph of conductivity plotted against volume of base added is approximately V shaped and the end-point is found by extrapolating the linear portions of the graph. The end-point is found from the point where the two lines cross.

PROCEDURE:-

Plug a conductivity sensor into the Vernier interface and insert the interface into the USB port on the computer. Select the Conductivity Titration file from the Experiment folder to set up the data logging system. Place the sensor into a beaker of distilled water until required.

Use a pipette to place 10.0 cm^3 of hydrochloric acid (approximately 0.1 mol dm^{-3}) in a clean 250 cm^3 beaker. Add approximately 100 cm^3 of distilled water and swirl gently to mix.

Place the conductivity sensor in the solution and check that the platinum electrodes are completely covered. Click on the COLLECT button on the computer screen. When the conductivity value is steady, click on the KEEP button and enter zero as the initial volume in the pop-up window.

From a burette add 1.0 cm^3 of the standard sodium hydroxide solution (0.10 mol dm^{-3}) and swirl the contents of the beaker or use the sensor as a stirring rod. Press KEEP when the conductivity reading is steady and enter the volume in the pop-up window

Continue, in this way, to add sodium hydroxide in 1.0 cm^3 portions and to record both the total volume added and the steady conductivity reading after each addition, until a total volume of 20 cm^3 has been added. End the data logging by pressing the STOP button

Remove the conductivity cell, rinse it with distilled water and then replace it in the beaker of distilled water. Discard the contents of the titration beaker and rinse the sensor with distilled water.

Set the printer to Landscape orientation and print the graph.

Export the data from the computer to your floppy disc, using CT1 as the file-name.

Repeat the experiment but use 10 cm^3 of ethanoic acid (approximately 0.1 mol dm^{-3}) instead of the hydrochloric acid. Export the data from this titration using the file-name CT2.

Fill a burette with ammonium hydroxide solution (approximately 0.1 mol dm^{-3}). Check that there is no bubble under the tap and that the meniscus is at the zero mark. Using a pipette, place 10.0 cm^3 of standard hydrochloric acid in a 250 cm^3 beaker and titrate the solution as described previously. Export the data as CT3.

Data Processing:-

The computer software has stored the conductivity data in files ready for further use. As this experiment requires two extrapolations of straight lines, a calculation of an end-point and the concentration of the “unknown” solution, the data have to be transferred to a spreadsheet.

Either create your own spreadsheet to display the data and determine the position of the end-point or use the Conductivity Titration spreadsheet.

Check that your data have been imported into the spreadsheet and remove any spurious data from a previous experiment (this should not normally be necessary). Print the spreadsheet and its associated graphs and determine the titration end points from the intersection points on the graphs.

Calculate the concentration of each of the acid concentrations.

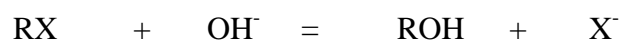
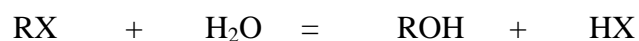
Discussion:-

- 1 Are there any significant advantages or disadvantages to this method of titration?
- 2 Would there be any advantage in making more measurements in the region of the end-point? Explain your answer.
- 3 Could this method be used for determining the concentration of weak acids in black- currant juice?

An Investigation into the Hydrolysis of 2-chloro- 2-methylpropane.

Introduction:-

Alkyl halides are important reagents in the synthesis of more complex organic molecules. They are much more reactive than the alkanes from which they are derived. They react with water and with hydroxide ions in a reaction termed hydrolysis to give an alcohol as the organic product:-



There are two mechanisms by which this process could occur. The S_N1 reaction proposes that the alkyl halide releases the inorganic anion in a slow step and the cation (or carbonium ion) left behind reacts rapidly with the water or hydroxide ion to produce the alcohol.

The S_N2 mechanism proposes that the carbon - halogen bond gets weaker as the oxygen of the water molecule or hydroxide ion approaches. As the bond between the carbon and oxygen becomes stronger, the halogen eventually breaks away as an anion and leaves behind a fully formed carbon-oxygen bond (with a hydrogen atom attached to the other oxygen bond).

These two processes can be distinguished by the way the rate of reaction is affected by changes in the concentrations of the reactants.

Procedure:-

Place 5 cm³ of propanone and 5 cm³ distilled water in a clean dry boiling tube. Mix well and then place the conductivity sensor in this solution, until required.

Measure accurately 5 cm³ of 2-chloro-2-methylpropane into a clean, dry conical flask and add 45 cm³ of propanone.

Measure accurately 5 cm³ of this solution and place it in a clean dry boiling tube. Measure accurately 5 cm³ of distilled water and add it to the alkyl halide solution. Mix well, insert the conductivity cell and begin collecting data at 10 second intervals. At some convenient point, record the temperature of the reaction mixture without disturbing the conductivity sensor.

After 40 minutes, stop the data logging and export the data to a file called tbca. Check that the printer is set to print in landscape format and then print the graph. Remove the conductivity sensor and stopper the boiling tube. Rinse the sensor with the propanone/water mixture.

Repeat the experiment, but using 2.5 cm³ of the propanone/water mixture, 2.5 cm³ of propanone and 5 cm³ of distilled water, collecting data at 15 second intervals. Export the data to a file called tbc b.

Record the “infinity” conductivity values of the reaction mixtures after an interval of not less than 90 minutes from the start of the experiments by placing the sensor in each solution in turn and logging the data for approximately 1 minute.. These values will need to be recorded in your laboratory note-book. Finally, rinse the sensor with propanone/water mixture and return it to its storage container.

Data Processing:-

In this experiment, the rate of reaction is being measured by logging the increasing conductivity due to the formation of a product. The rate of increase is quite rapid at the start of the reaction, when the concentration of the reactants is at its greatest but decreases as the reactants become used up. The reaction is complete only after an “infinitely long” period of time.

There are two methods of avoiding this difficulty - one is to estimate an infinity value, based on the shape of the graph and how well the curve fit option behaves (“modelling”), the other is to measure the conductivity at a point in time when there is very little further change of conductivity to be observed.

When a reaction involving the alkyl halide, RX, is a first order reaction, the rate of reaction is given by the equation –

$$rate = -\frac{d[RX]}{dt} = k[RX]$$

This can be rearranged to give -

$$-\frac{d[RX]}{[RX]} = kdt$$

and integrated to give -

$$-\ln[RX] = kt + C$$

When $t = 0$, the integration constant C is found to be $-\ln[RX]_0$ and taking antilogs of both sides leads to -

$$[RX] = [RX]_0 \exp(-kt)$$

Since there is a 1:1 relation between the concentration of RX used up and the concentration of HX formed, it follows that -

$$[HX] = [RX]_0 (1 - \exp(-kt))$$

Select the Hydrolysis spreadsheet and enter the initial and “infinity” values of conductivity. Check that your data have been imported into the spreadsheet and then print both the data tables and the graphs.

Change the entries in the “time” and “conductivity” columns from tbca.txt to tcbb.txt in order to import the second set of data. Follow the spreadsheet instructions from this point to open the second data file. After printing the second set of data, close the spreadsheet **without** saving the changes.

Results:-

A linear graph indicates that the reaction is 1st order with respect to 2-chloro 2-methylpropane. This means that the alkyl halide is involved in the slowest step in the reaction mechanism.

Investigating the Bromine – Propanone Reaction

INTRODUCTION

The reaction between bromine and propanone is similar to the reaction between iodine and propanone. It is catalysed by hydrogen ions and can be followed colorimetrically, by measuring the decreasing absorbance at 430nm or 470 nm, due to bromine. The rate of reaction may also be followed by removing samples at intervals, treating with a small excess of potassium iodide and then titrating with standard sodium thiosulphate solution.

Bromine is an unpleasantly smelling, dark red liquid which must always be treated with great caution. Bottles of bromine should not be opened in the open laboratory but only in an efficient fume chamber. For this experiment, bromine is more conveniently supplied as a saturated solution which must also be kept in the fume cupboard. Further, all reaction mixtures must be prepared and the cuvettes filled in the fume cupboard.

PROCEDURE

Make a note of the laboratory temperature.

Connect the interface and colorimeter to the computer before preparing any of the reaction mixtures. Set the colorimeter to record absorbance at 430 nm and to collect data at intervals of 10 seconds. Set the printer to print graphs in landscape format. Save the settings in your network file using the file name “Blank Setup”. **If, at any time you wish to save the display of graph and table, use the Save As option – do not use the Save option as this will write over anything you have previously saved.**

Prepare a reaction mixture by placing the following volumes of reagents in a small, (eg 50 cm³) dry beaker (use a burette for accurate measurement of the volumes and add the reactants in the order given):

10 cm³ 2 mol dm⁻³ hydrochloric acid
10 cm³ of distilled water
6 cm³ of saturated bromine water
1 cm³ of propanone

Swirl the contents of the beaker to ensure good mixing and then three-quarters fill a cuvette (colorimeter cell) with the solution. Place the cuvette in the colorimeter, close the lid and click on the start button. Collect data for a period of 4 - 5 minutes. Click on the Stop button to end the data logging. Print the graph and then save the data by selecting Export Data from the File menu. Click on Run 1 which appears in the pop-up window and give the data file a suitable name. After you have saved the data, select Delete Last Data Run from the Experiment drop-down menu to clear the graph and data table.

Remove the cuvette and discard the solution to a waste jar. Rinse the cuvette and dry it with a paper tissue.

Repeat the above procedure with a range of mixtures in which you vary the concentration of –

- a) propanone
- b) hydrochloric acid
- c) bromine water

by varying the volume of only one reactant at a time and keeping the total volume constant (only the volume of water may be altered to keep the total volume constant).

When all the different mixtures have been measured, prepare the following mixtures and measure their absorbances - this “one-point calibration” will allow you to convert absorbance readings into bromine concentrations.

DATA PROCESSING

Data processing may be done by using the Curve Fit option on each graph as soon as the data collection has ended – use either a linear fit, if the points appear on a straight line or a polynomial fit if the line is curved – or the data may be entered into an Excel spreadsheet.

Calculate the composition of each reaction mixture and convert the slope of the corresponding graph into a rate of reaction.

Determination of the Enthalpy of Vaporisation of Water

Introduction:-

The normal boiling point of a liquid is the temperature at which its equilibrium vapour pressure is equal to the prevailing atmospheric pressure (101.35 kPa). It is a well-known fact that water boils at a much lower temperature at the top of a mountain where the atmospheric pressure is much less than at sea level. The change in boiling point caused by the change in pressure is related to the enthalpy of vaporisation by the equation –

$$\ln P = - \frac{\Delta H_{\text{vaporisation}}}{RT}$$

By measuring the boiling point of a liquid at a number of different pressures, it is possible, therefore, to determine the value of the enthalpy of vaporisation by plotting the natural log of the pressure (ln P) against the reciprocal of the absolute temperature. The slope of the resulting graph gives the value of $\Delta H_{\text{vaporisation}}/R$.

Procedure:-

The Quickfit flask to be used in this experiment must be carefully checked to ensure that there are no cracks or star fractures which could cause the flask to implode under the reduced pressure conditions of this experiment. Once the apparatus has been set up, it must be checked by the teacher in charge that it is safe to continue.

Place approximately 150 cm³ of water in a 500 cm³ round bottomed, 3 necked Quickfit flask and support the flask either in a heating mantle or on an electric hot-plate. Insert a reflux condenser (fitted with a stopper) into the centre neck and a Vernier temperature probe into one of the other necks, if possible in such a way that refluxing liquid falls upon the sensor. Support the condenser in a clamp stand. Insert a glass T-piece into the remaining neck and attach a Vernier pressure sensor to one of

the T-piece limbs. Connect the other limb of the T-piece to a vacuum pump via a straight tap-piece.

Launch the LoggerPro software and open the Enthalpy of Vaporisation file to set up the data collection. Click on the **Collect** button to display the current pressure and temperature.

Place a safety screen in front of the apparatus. **Before proceeding further, have the assembly checked for safety and also that it can hold the vacuum.**

With the tap closed, switch on the vacuum pump and then carefully open the tap to reduce the pressure inside the flask. When the pressure has fallen to approximately 30 kPa, close the tap and begin heating the liquid in the flask. When both temperature and pressure readings are steady, click on the **Keep** button to record the data.

Open the tap slightly to increase the pressure in the flask by approximately 10 kPa. When the temperature has adjusted to the new pressure and is steady, click on the **Keep** button to record the data.

Repeat this procedure until the pressure has reached normal atmospheric pressure (approximately 100 kPa). After recording the last data, click on the **Stop** button to end the data logging process.

Allow the flask and its contents to cool before taking the apparatus apart and disposing of the hot water.

Data Analysis:-

When the natural log of the vapour pressure ($\ln P$) is plotted against $1/T$ (the absolute temperature, not the Celsius temperature) a straight line results, the slope of which is given by –

$$\text{Slope} = -\Delta H^{\circ}/R$$

Since the value of R is $8.314 \text{ J K}^{-1} \text{ mol}^{-1}$, the enthalpy of vaporisation can be calculated from –

$$\Delta H^{\circ} = -8.314 \times \text{slope} \text{ J K}^{-1} \text{ mol}^{-1}$$

Other liquids which may be studied in the same way include –

Compound	Boiling Point/$^{\circ}\text{C}$
Methanol	65
Ethanol	78
Ethanoic Acid	118
Propanone	57
Propan-2-ol	84
sec-Butanol	99
butan-1-ol	118
iso-Butanol	108
tert-Butanol	83
Cyclohexane	81

Trouton's Rule states that, for a non-associated liquid, the ratio of the enthalpy of vaporisation to the normal boiling point (absolute temperature) is approximately $90 \text{ J K}^{-1} \text{ mol}^{-1}$.

$$\Delta H_{\text{vap}}/T_{\text{b pt}} \quad 90 \text{ J K}^{-1} \text{ mol}^{-1}$$

Look up the enthalpies of vaporisation of the above compounds and calculate Trouton's constant for each one.

Which of the compounds obey Trouton's Rule?

Why do the other compounds not obey this rule?

Can you suggest another name for Trouton's Constant?

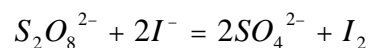
Appendix:-

Compound	Enthalpy of Vaporisation/ J mol^{-1}	Trouton's Constant
methanol	38512	114.0
ethanol	41940	119.4
ethanoic acid	40278	103.0
propanone	31147	94.1
propan-2-ol	42273	118.4
sec butanol	45254	121.5
isobutanol	46214	121.3
tert-butanol	43465	122.1
butanol	47548	121.8
cyclohexane	30915	87.4
hexane	31455	92.1
water	39710	106.5

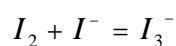
Determination of the Orders of Reaction in the Peroxodisulphate/Iodide Reaction.

Introduction:-

This reaction was one of the first inorganic reactions to be studied with the intention of discovering how reactant concentrations affect the rate of the reaction. It is not as simple as it appears from the equation –



because the iodine combines with some of the remaining iodide ion to form the complex tri-iodide ion -



This problem can be avoided by allowing the reaction to produce only a very small amount of iodine. This method is called the initial rate method. Its chief advantage is that it reduces the effect of interfering reactions between a product and the remaining reactants.

In order that this method may be used, there has to be a very sensitive method of measuring the concentration of either a reactant or a product. Iodine dissolves in the presence of potassium iodide to give a very dark brown solution. The colour is so intense that even very small concentrations of iodine can easily be detected.

Procedure:-

1 Preparation and Calibration

Peroxodisulphate solutions are not very stable and should be made just before use. Place 2.70 g of potassium peroxodisulphate in a clean dry beaker and dissolve it in 100 cm³ of distilled water. Transfer the solution to a 250 cm³ graduated flask and rinse the beaker several times with distilled water. Transfer the rinses to the flask and make up to the mark with distilled water. Record the temperature of the solution.

Potassium iodide solutions tend to oxidise slowly - if the solution is pale yellow/brown a fresh solution must be prepared from 3.3 g of potassium iodide dissolved in distilled water and made up to a final volume of 100 cm³.

Insert the colorimeter into the Vernier interface and connect the interface to the USB computer socket. Set the colorimeter to record absorbance using the blue light (468 nm) source at 5 second intervals. Place 1 cm³ of 0.05 mol dm⁻³ iodine solution in a 10 cm³ graduated flask and make up to the mark with distilled water.

Mix thoroughly and three-quarters fill the colorimeter cell with the solution. Record the absorbance of this solution by pressing the on-screen COLLECT button and allowing the software to collect data for approximately one minute. Remove the cell, discard the solution and rinse the cell with distilled water. Make a note of the absorbance value and the exact concentration of the iodine solution and then delete the data.

2 Determination of the Order of Reaction for Peroxodisulphate

In a clean dry beaker place 5 cm³ of potassium peroxodisulphate solution, 5 cm³ of water and 5 cm³ of potassium iodide solution. Swirl the mixture to obtain a uniform solution and transfer approximately 3 cm³ rapidly to the colorimeter cell. Place the cell in the colorimeter and click on COLLECT to commence data logging.

When sufficient data have been collected, click on the STOP button to end the data logging and print the graph, together with the appropriate curve-fit applied. Discard the contents of the cell and rinse it with distilled water. Dry the cell with a paper tissue.

In a clean dry beaker place 10 cm³ of potassium peroxodisulphate solution and add 5 cm³ of potassium iodide solution. Swirl rapidly to mix and then transfer approximately 3 cm³ of the solution to the 1 cm cell. Collect data, as before, and print the graph, after applying the appropriate curve-fit. Discard the contents of the cell, rinse it with distilled water and dry it with a paper tissue. Clear the last set of data from the screen.

3 Determination of the Order of Reaction for Iodide

In a clean dry beaker place 5 cm³ of potassium peroxodisulphate solution and add 10 cm³ of potassium iodide solution. Mix the solutions by rapid swirling and then transfer to the cell. Collect data as before and print the graph, after applying the appropriate curve-fit.

Results:-

1 Calibration

Calculate the concentration of the iodine solution used in the calibration of the colorimeter and the factor which will convert absorbance into concentration.

2 Determination of the Order of Reaction for Peroxodisulphate

What effect does doubling the concentration of peroxodisulphate have on the rate of reaction?

What is the order of reaction for peroxodisulphate ?

3 Determination of the Order of Reaction for Iodide

What effect does doubling the concentration of iodide have on the rate of reaction?

What is the order of reaction for iodide ?

4 Determination of the Rate Constant for the Reaction

Use the calibration factor to convert the slopes of the graphs into reaction rates (in mol dm⁻³ s⁻¹) and calculate the concentrations of the reactants in each of the three reaction mixtures.

Use the rate equation -

$$rate = k[S_2O_8^{2-}]^x[I^-]^y$$

where **x** and **y** are the orders of reaction, to calculate the value of the rate constant from each set of data.

Determination of Orders of Reaction and Rate Constant ~ (Propanone/Iodine Reaction)

Introduction:-

This reaction was first studied one hundred years ago, in order to discover how the halogens reacted with hydrocarbons to form substitution products. The hydrocarbons, themselves, were too unreactive for the techniques of the time and the reactions of alcohols with halogens were complicated because oxidation took place as well as substitution.

For many years, the preferred method of investigating the rate of chemical reactions was to remove a sample, stop the reaction (if possible) and then do a titration of some kind to determine how much of a product had been formed or how much of a reactant was still left. This method had several disadvantages and this experiment uses a physical method of measuring the concentration of a reactant, without having to remove a sample from the reacting solution.

The reaction between propanone and iodine is represented by the equation –



The equation indicates that propanone and iodine react in a 1:1 ratio and that hydrogen ions and iodide ions are formed in addition to the main product iodopropanone. It does not give any indication of how fast the reaction occurs or how the rate of the reaction is affected by the concentrations of reactants and products. This information can only be obtained by performing experiments.

Procedure:-

Prepare a 2.0 mol dm^{-3} solution of propanone in water by weighing 58 g (ca 46 cm^3) of propanone into a 500 cm^3 volumetric flask and making up to the mark with distilled water.

Insert a colorimeter into the Vernier interface connect the interface to the computer USB port. Three-quarters fill a colorimeter cell with distilled water, place it in the colorimeter and press the Calibrate button, to establish the value of zero absorbance.

From the Experiment folder, select the Propanone file to load the appropriate control and display settings for this experiment. Click on Data on the title bar and select Data Collection to check that data will be collected at 5 second intervals.

In a dry boiling tube, place 2 cm^3 of 0.05 mol dm^{-3} iodine solution, 2 cm^3 of 2 mol dm^{-3} hydrochloric acid and 2 cm^3 of distilled water, all volumes accurately measured by pipette or from a burette. Add 2 cm^3 of propanone solution, also by pipette or from a burette, and mix thoroughly. Three-quarters fill the colorimeter cell with this mixture, close the lid and click on COLLECT to commence data logging.

End the collection of data when the absorbance has decreased by one quarter of its initial value, or after two minutes, whichever is the shorter period of time by clicking on the STOP button. Export the data to a file named Run1. Discard the solution in the cell and rinse with distilled water.

Prepare the following mixtures, but add the propanone solution only when you are ready to place the mixture in the colorimeter:-

Run No.	Iodine/ cm^3	Acid/ cm^3	Water/ cm^3	Propanone/ cm^3
2	2.0	4.0	0.0	2.0
3	2.0	2.0	0.0	4.0
4	1.0	2.0	2.0	2.0

Rinse the colorimeter cell with some of the reaction mixture and then three-quarters fill the cell. Collect data as before and export each data set to a named file on disc. When all the data logging has been completed, remove the cell and rinse it with distilled water.

Finally, place 2.0 cm^3 of 0.05 mol dm^{-3} iodine solution in a clean dry boiling tube and add 6 cm^3 of distilled water. Mix thoroughly and then rinse the colorimeter cell with the solution, before three-quarters filling the cell. Measure the absorbance of this solution by collecting data for a period of approximately 1 minute. Record the value of this absorbance as A^0 .

Data Processing:-

Select **Page Setup** from the File menu and check that the printer is set to print in Landscape orientation. Maximise the graph window and then select **Print** from the File menu.

Measure the slope of each graph of Absorbance vs time data.

The Absorbance (A^0) of the diluted iodine solution is needed to convert the value of the slope into a rate of reaction:-

$$2.0\text{ cm}^3\text{ of }0.05\text{ mol dm}^{-3}\text{ iodine} = 8\text{ cm}^3\text{ of }x\text{ mol dm}^{-3}\text{ iodine}$$

therefore $x = 0.0125 \text{ mol dm}^{-3} \text{ iodine}$

$$\text{Absorbance} = A^0 = k [\text{I}_2]$$

$$k = A^0/[\text{I}_2]$$

To convert a slope (with units of s^{-1}) to rate of reaction (units of $\text{mol dm}^{-3} \text{ s}^{-1}$), divide by the constant k (units of $\text{dm}^{-3} \text{ mol}^{-1}$).

Compare the rates of reaction from Run 1 and Run 2 to observe the effect of doubling the concentration of hydrochloric acid.

Compare the rates of reaction from Run 1 and Run 3 to observe the effect of doubling the concentration of propanone.

Compare the rates of reaction from Run 1 and Run 2 to observe the effect of halving the concentration of iodine.

Calculate the concentration of each reactant in each of the four reaction mixtures and then use the following equation to calculate the value of the rate constant:-

$$\text{Rate of reaction} = k [\text{Propanone}]^x [\text{H}^+]^y [\text{Iodine}]^z$$

Points to consider:-

How is the rate equation different from what you might have expected from the reaction equation?

Why does the concentration of hydrogen ions affect the rate of reaction?

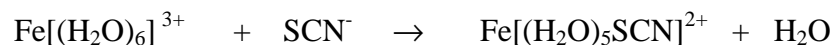
Does the reaction take place as a result of a simple collision between propane and iodine molecules?

This method of having all the reactants except one in very large concentrations compared with one selected reactant is called the isolation method. Why does it work and what are its advantages/disadvantages?

Determination of an Equilibrium Constant (FeSCN²⁺)

Introduction:-

The reaction between iron(III) ions and thiocyanate ions has been studied for many years and there are many Internet sites which use this reaction to illustrate Le Chatelier' Principle. The thiocyanate ion is colourless and the iron(III) ion is almost colourless in dilute solutions but the complex formed when they react is an intense reddish brown colour –



This makes it an ideal system to examine using a colorimeter to measure the increasing absorbance of the complex ion and from this determine the value of the equilibrium constant.

The information gathered in this experiment comprises the absorbances (A) of the solutions and the amounts of iron(III) ([Fe]) and thiocyanate ([SCN]) ions used. The absorbance of a solution is directly proportional to the concentration of the complex ion –

$$A = k [\text{FeSCN}^{2+}]$$

from which –

$$[\text{FeSCN}^{2+}] = A/k$$

The initial concentrations of the reactants are –

$$[\text{Fe}^{3+}] = C_f$$

$$[\text{SCN}^-] = C_s$$

The equilibrium constant is written as –

$$K = \frac{[FeSCN^{2+}]}{[Fe^{3+}][SCN^{-}]}$$

which, after substituting the above symbols, becomes –

$$K = \frac{A/k}{(C_f - A/k)(C_s - A/k)}$$

Although it is quite easy to find the value of K if you know the initial and equilibrium concentrations of the reactants, it is much more difficult if you don't know how much complex ion has been formed. The absorbance of the complex ion is proportional to its concentration but the value of the constant k has not been determined. However, because the concentrations of reactants and product are very small, it is possible to use some advanced mathematics (series inversion) to change the last equation into the form –

$$A/k = \frac{C_f C_s}{k} + \frac{1}{kK}$$

This shows that a plot of A/k vs $C_f C_s$ will be linear with a slope having the value of $1/k$ and an intercept having the value of $1/kK$. So, it is possible to determine the value of the equilibrium constant from the equation –

$$K = \text{slope/intercept}$$

Procedure:-

Prepare a fresh solution of iron(III) nitrate by dissolving 4.04 g of the hydrated salt ($Fe(NO_3)_3 \cdot 9H_2O$) in 25 cm^3 of 2 mol dm^{-3} nitric acid and making up to a final volume of 100 cm^3 with distilled water to give a concentration of 0.10 mol dm^{-3} .

Prepare a solution of potassium thiocyanate by dissolving 0.972 g of the salt in 25 cm^3 of distilled water and making up to a final volume of 100 cm^3 . (If ammonium thiocyanate is used, the mass of salt should be 0.76 g). The concentration of this solution will be 0.10 mol dm^{-3} . Transfer, by pipette, 10.0 cm^3 of this solution to a 500 cm^3 graduated flask and make up to the mark with distilled water. It is important that the flask is thoroughly shaken throughout the addition of water to ensure a uniform solution. The concentration of this solution will then be $0.002 \text{ mol dm}^{-3}$. Finally, transfer by pipette, 10 cm^3 of the $0.002 \text{ mol dm}^{-3}$ solution to a 100 cm^3 volumetric flask, add 25 cm^3 of 2 mol dm^{-3} nitric acid and make up to the mark with distilled water.

Plug the colorimeter into the Vernier interface and connect the interface to the computer USB port. Click on **File** and select **FeSCN** from the Experiments folder, to load the colorimeter settings for this experiment. Calibrate the colorimeter by placing a cell containing distilled water in the colorimeter and pressing the green Calibrate button. After calibration, empty the cell and use a paper tissue to remove as much of the remaining water as possible.

Transfer all of the $0.002 \text{ mol dm}^{-3}$ thiocyanate solution to a clean dry 250 cm^3 beaker. Add 1.0 cm^3 of the iron(III) nitrate solution from a burette and swirl the contents of

the beaker to ensure good mixing (the volume need not be exactly 1.0 cm³ but it must be accurately known). Carefully transfer sufficient of the pale coloured solution to fill the colorimeter cell to approximately three-quarters. Place the cell in the colorimeter and measure the absorbance. When the pop-up window appears, enter the volume of the iron(III) solution which has been added.

Return the solution to the beaker and add a further 1.0 cm³ of iron(III) solution from the burette and swirl the solution. Rinse the cell with this solution, return the solution to the beaker, swirl again and then three quarters fill the cell. Measure the absorbance and enter the volume of iron(III) solution used, as before. Return the solution to the beaker as before.

Continue, in this manner, to add further quantities of iron(III) solution and to record both the absorbance of the resulting solutions and the volumes of iron(III) solution used.

Finally, rinse the colorimeter cell with distilled water and return it, with the colorimeter, to its storage box. Discard all the solutions.

Data Processing:-

Store the data in a file called **fescn** (select Export Data from the File menu) and follow the on-screen instructions). Set the printer to Landscape orientation and then select Print to obtain a copy of the graph.

As there is quite a large amount of repetitive calculation involved in this experiment, it is appropriate to use a spreadsheet. The calculations which have to be made are:-

The concentrations of [Fe³⁺] and [SCN⁻] for each mixture, as the volume is constantly increasing.

The sum of the concentrations of [Fe³⁺] and [SCN⁻] for each mixture.

The product of the concentrations of [Fe³⁺] and [SCN⁻] for each mixture divided by the corresponding absorbances.

The graph to be drawn is of $([\text{Fe}^{3+}] + [\text{SCN}^-])/A$ vs $([\text{Fe}^{3+}] + [\text{SCN}^-])$.

To link your spreadsheet to your data file, use the following entry to transfer the Absorbance values to the first of the cells in the Absorbance column and copy it down to the last value -

= 'A:\[fescn.txt]fescn'!A3

(this assumes that you have stored your data on a floppy disc in the A:\ drive). Similarly, to transfer the volumes of Fe³⁺ solutions used, use -

= 'A:\[fescn.txt]fescn'!B3

in exactly the same way.

An Excel spreadsheet (FESCN) is available for comparison.

References:-

Iron Thiocyanate Revisited, C L Cobb, G A Love, J Chem Ed, 75, 90 – 92, (1998)

Demonstrating Job's Method, W R Carmody, J Chem Ed, 41, 613 – 615, (1964)

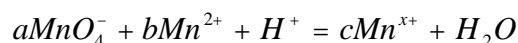
Investigating a Redox Reaction

Introduction:-

Manganese in its highest oxidation state (Mn(VII)) is a very powerful oxidising agent, especially in strongly acid solution:-



In this experiment it oxidises the commonest oxidation of manganese (Mn(II)) to a higher oxidation state:-



The aim of this experiment is to determine the values of a, b and c.

Materials Required:-

0.004 mol dm⁻³ manganese(II) sulphate, made up in 6 mol dm⁻³ sulphuric acid

0.01 mol dm⁻³ potassium permanganate, made up in distilled water.

Procedure:-

Set the colorimeter to record absorbance at 470 nm (blue source) and calibrate with the manganese(II) solution as the blank reagent. Select the REDOX file from the Experiment folder to set up the data collection with keyboard entries.

Place 10 cm³ of manganese(II) solution, accurately measured by pipette, into each of eight clean, dry 100 cm³ beakers. Prepare the following reaction mixtures, measuring each reagent as accurately as possible:-

KMnO ₄ /cm ³	0.5	1.0	1.5	2.0	2.5	3.0	3.5	4.0
H ₂ O/cm ³	9.5	9.0	8.5	8.0	7.5	7.0	6.5	6.0

Swirl each beaker to ensure good mixing and then record the absorbance of each solution in turn, entering the numbers 1 to 8 to identify the solutions.

To measure the absorbance of a solution, first rinse the empty cell with 2- 3 cm³ of the solution. Discard this solution and then three-quarters fill the cell with solution.

Measure the absorbance and then discard the solution. After measuring the absorbance of the last solution, rinse the cell several times with distilled water.

Plot the raw data (Absorbance vs Solution Number) and print both the graph and the data table. Export the data to a named file on the a: drive.

Data Processing and Interpretation:-

The blank solution contains only manganese(II), which does not absorb visible radiation and therefore appears colourless. There is only one coloured product in this reaction and this absorbs the 465 nm radiation very strongly.

When all of the manganese(II) has been oxidised to this new state, the excess permanganate will also absorb at this wavelength but not as strongly as the product.

Create a spreadsheet which will display the concentrations of the reagents used in the experiment and the composition of the reaction mixtures. Import the absorbance data and calculate the ratio of $[\text{Mn(VII)}]/[\text{Mn(II)}]$ for each solution.

Plot the absorbance of the solution against the ratio of $[\text{MnO}_4]/[\text{Mn}^{2+}]$ and draw the best straight lines through the linear portions of the graph. The point where the lines intersect gives the ratio in which the permanganate and manganese(II) react.

- 1 What is the reacting ratio of manganese(VII) to manganese(II) ?
- 2 What is the product of the reaction ?
- 3 Does the product have oxidising or reducing properties?

To determine the molar Mass of a Metal by Electrodeposition

INTRODUCTION:-

Faraday's Laws of Electrolysis relate the amount of chemical change taking place at the electrodes to the size of the current and the time for which the current flows. The product of current multiplied by time is the amount of electrical charge which has been transferred at the electrodes as the redox reactions take place. The amount of charge associated with 1 mole of electrons is a quantity known as the Faraday and amounts to 96500 Coulombs.

In this experiment, the data logger monitors the current flowing through the electrolysis circuit during a period long enough to produce weighable amounts of copper. When the current is plotted against time, the area under the graph gives the amount of charge that has been transferred at each electrode. From the amount of charge associated with the measured mass of the copper deposited or dissolved, it is possible to determine the molar mass of the metal, always remembering that the copper ion carries two positive charges.

PROCEDURE:-

Launch the LoggerPro software and load the Molar Mass file to set up the data logging parameters.

Connect the white BT terminal of the current probe computer lead to the interface.

Cut two strips of copper foil, approximately 8cm x 2 cm, and clean them by immersing them for 10 – 20 seconds in dilute (2 mol dm⁻³ nitric acid). Rinse the strips with water and then with propanone or ethanol and allow them to dry. Bend one corner of one strip, so that it can be distinguished from the other. Weigh each strip and record its mass.

Place 50 cm³ of 1.0 mol dm⁻³ copper sulphate solution in a 100 cm³ beaker and dip both electrodes into the solution. Support the electrodes so that they do not touch. Connect one electrode to the positive terminal of the power supply and the other, to the positive terminal on the current probe. Connect the negative terminal of the current probe to the negative terminal of the power supply..

Have your apparatus checked by your teacher/teaching assistant before proceeding further with the experiment.

Click on the Collect button on the computer screen and then switch on the power supply to the electrolysis cell. Adjust the voltage to give a current between 200 mA and 500 mA (0.20 – 0.50 A) and leave the apparatus for a period 20 minutes. At the end of this period, when the data logging will stop automatically, switch off the power supply to the cell and remove the electrodes. Rinse both electrodes carefully with distilled water and then with ethanol or propanone. Take particular care with the cathode as the copper deposited on the electrode may have a tendency to fall off. Leave the electrodes to dry and then weigh each one separately.

DATA PROCESSING:-

Follow the on-screen instructions to calculate the area under the graph by integration and to place the value on the graph in a text annotation. Print the graph and then use the data as follows:-

Initial mass of cathode - w_1 g
Final mass of cathode - w_2 g
Change in mass - $w_2 - w_1$ g

Area under the graph - C Coulombs
No. of charges on the ion - 2

C Coulombs deposit $w_2 - w_1$ g of Copper

1 mole of Cu^{2+} ions require 2 moles of electrons (= 2 x 96500
Coulombs)
in order to be deposited as metal

therefore

2 x 96500 C will deposit $\frac{(w_2 - w_1)96500 \times 2}{C}$ g of copper

A similar calculation using the decrease in mass of the anode will lead to a similar value for the mass of copper which dissolves to furnish 2 moles of electrons.

Conductivity of Weak Electrolytes - the Ostwald Dilution Law

Introduction:-

Weak electrolytes are solutes which do not dissociate into ions to any great extent. Many organic acids and bases fall into this classification. A typical weak monobasic acid, represented by HX, ionises to a small extent –



for which the (concentration) equilibrium constant is written as –

$$(1) \quad K_a = \frac{[\text{H}^+][\text{X}^-]}{[\text{HX}]}$$

In an acid solution, with an initial concentration of $c \text{ mol dm}^{-3}$ of the weak acid, at equilibrium there will be a certain fraction, a , which has ionised. The concentrations of hydrogen ion, anion and unionised acid are then given by –

$$[\text{H}^+] = \alpha c$$

$$[\text{X}^-] = \alpha c$$

$$[\text{HX}] = (1 - \alpha)c$$

When these are substituted in the equilibrium constant definition (1), it becomes –

$$(2) \quad K_a = \frac{a^2 c}{(1 - a)}$$

If the electrolyte is very weak, the degree of ionisation (α) will be very small and $(1 - \alpha)$ will not be very different from 1. Under these conditions, equation (2) simplifies to –

$$(3) \quad K_a = a^2 c$$

and re-arranges to give –

$$(4) \quad a = \sqrt{\frac{K_a}{c}}$$

This is one form of the Ostwald Dilution Law which states that the degree of ionisation of a weak electrolyte is inversely proportional to the concentration (or, since dilution is the inverse of concentration, the degree of ionisation is directly proportional to the dilution).

As the solution becomes more dilute, more and more of the weak electrolyte ionises and eventually, in an infinitely dilute solution, it becomes completely ionised. In this infinitely dilute solution, one mole of the electrolyte has a conductivity represented by

Λ^∞ . In less dilute solutions, the molar conductivity is smaller (because of the lesser degree of ionisation) and is represented by Λ .

The degree of ionisation can be calculated from the ratio –

$$(5) \quad \alpha = \frac{\Lambda}{\Lambda^\infty}$$

When this is substituted in (2), the result is –

$$K_a = \frac{(\Lambda / \Lambda^\infty)^2 c}{(1 - \Lambda / \Lambda^\infty)}$$

which rearranges to give

$$\frac{1}{\Lambda} = \frac{1}{\Lambda^\infty} + \frac{\Lambda c}{K \Lambda^{\infty 2}}$$

The molar conductivities can be calculated from the conductivity (κ) and concentration (c) of each solution –

$$\Lambda = \frac{\kappa}{c}$$

Procedure:-

The success of this experiment depends on accurate measurements and extremely clean glassware.

Standardise a solution of ethanoic acid, which is approximately 0.1 mol dm^{-3} by titration against standard $0.100 \text{ mol dm}^{-3}$ sodium hydroxide, using phenolphthalein as indicator.

Make the following dilutions by accurately dispensing the stated volume of acid into a clean volumetric flask and making up to the mark with distilled/deionised water. Thoroughly mix the contents of each flask before making any measurements.

Volume of Acid Solution/cm ³	Concentration/mol dm ⁻³	Final Volume of Solution/cm ³	Final Concentration/mol dm ⁻³
50.0	0.100	100.0	0.0500
25.0	0.100	100.0	0.0250
10.0	0.100	100.0	0.0100
5.0	0.100	100.0	0.00500
10.0	0.0100	100.0	0.000100

Attach a Vernier conductivity sensor to the interface and connect the interface to the computer. Select *Open* from the *File* drop-down menu and select *Ostwald* from the list of available experiments.

Rinse the conductivity probe with 0.10 mol dm^{-3} of ethanoic acid and then immerse the probe in 20 cm^3 of 0.10 mol dm^{-3} ethanoic acid in a clean dry 50 cm^3 beaker.

Record the conductivity of the solution and enter the concentration in the pop-up window.

Rinse the conductivity probe twice with the next solution and then measure the conductivity of the solution, as above. Proceed in this way with the remaining solutions. Finally, rinse the probe twice with distilled/deionised water, record the conductivity of the water and return the probe to its storage container.

Data Processing:-

The Vernier conductivity probe measures conductivity ($\mu\text{S cm}^{-1}$) directly as it is already calibrated and has a cell constant of 1 cm^{-1} .

Several conversions to SI units have to be made –

Conductivity in $\mu\text{S cm}^{-1}$ is converted to S m^{-1}

Concentration in mol dm^{-3} is converted to mol m^{-3}

The intercept of the graph resulting from plotting $1/\Lambda$ against Λc gives the value of $1/\Lambda^\infty$ its reciprocal gives the limiting molar conductivity (Λ^∞ - at “infinite dilution”).

The slope of the graph gives the value of $1/(K_a \Lambda^\infty{}^2)$.

To obtain the value of the dissociation constant, take the reciprocal of the slope and multiply it by the square of the value of the intercept. This will give the value of K_a in mol m^{-3} units. Divide this answer by a factor of 1000 to convert to mol dm^{-3} units.

Reference:-

**D A MacInnes, T Shedlovsky, JACS, 34, 1429 (1932)
Electrode Potential Changes in a Redox Reaction**

Introduction:-

In a redox reaction, the changing concentrations of the two oxidation states of either the reducing agent (iron(II) ions in this case) or the oxidising agent (manganate(VII) ions in this experiment) can be followed by placing a platinum electrode in the solution, as part of an electrochemical cell. The platinum electrode does not take part in the redox reaction – it behaves in much the same way as a pH electrode does in an acid/base titration.

Procedure:-

Prepare a standard solution of ammonium iron(II) sulphate by dissolving 3.92 g of the salt in 50 cm³ of warm 1 mol dm⁻³ sulphuric acid and making up to a final volume of 100 cm³ with distilled water. Transfer 25.0 cm³ of this solution to a clean beaker and add an equal volume of 1 mol dm⁻³ sulphuric acid.

Fill a clean, dry burette with a potassium manganate(VII) solution which is approximately 0.02 mol dm⁻³. Check that there is no air bubble beneath the tap and set the initial reading to 0.0 cm³.

Place the ORP (oxidation/reduction potential) sensor in the solution and switch on the magnetic stirrer if available. Load the experiment Redox Changes from the Experiments folder on your computer and follow the initial on-screen prompts.

When read to start the titration, click on the Collect button (on screen) and add 4 cm³ of manganate(VII) solution from the burette. Record the potential and the volume of solution added and continue in this way until 20 cm³ of solution has been added. Continue by adding manganate(VII) in small volumes until the slope of the graph just begins to increase and then add the solution drop by drop until the slope decreases again.

Discard the solution in the beaker and repeat the titration twice more to obtain a pair of end-points which agree to within 0.1 cm³

Data Processing:-

Follow the on-screen prompts to print titration curves and to store the data for later pasting into your practical book.

Investigate what happens to the graphs when you use the first and second derivative options. Does this help in determining where the end-point of the titration lies?

Estimate as accurately as you can the cell potential when the titration is at exactly its half-way point. The cell potential is controlled by the equation –

$$E_{cell} = E_{redox} - E_{ref}$$

The reference electrode has a potential of 0.222V at 298 K. The potential of the redox electrode is therefore given by –

$$E_{redox} = E_{cell} + E_{ref}$$

The potential of the redox electrode is controlled by the Nernst equation –

$$E_{redox} = E^{\circ} + \frac{RT}{nF} \ln \frac{[Oxidised]}{[Reduced]}$$

What is the special significance of the potential of the redox electrode at the half-way point ?

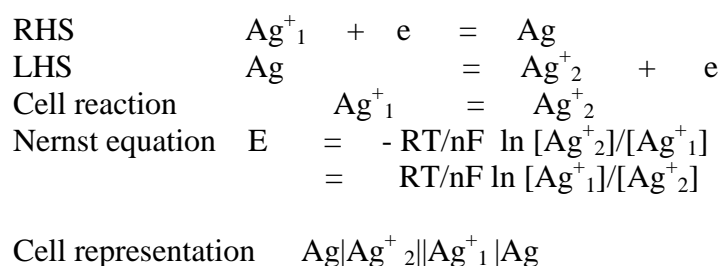
Electrochemical Determination of the Solubility of a Sparingly Soluble Salt

INTRODUCTION:-

The EMF of a galvanic cell is governed by the Nernst equation –

$$E = E^{\circ} - \frac{RT}{nF} \log_e \frac{\sum [\text{products}]}{\sum [\text{reactants}]}$$

Concentration cells represent a special case of this equation since these cells have the same half-cell reactions and the value of E° is zero. The cell EMF is due to the difference in concentrations and the cell reaction is effectively the dilution of the more concentrated solution:-



In this experiment, the left hand side of the cell consists of a silver electrode immersed in a saturated solution of silver chloride. The silver ion concentration in this half cell is determined by the value of the solubility product of silver chloride. The right hand side of the cell comprises a silver electrode immersed in solutions of silver nitrate in the range 0.1 mol dm^{-3} to $0.001 \text{ mol dm}^{-3}$.

The Nernst equation for a concentration cell can also be written as –

$$E = \frac{RT}{nF} \ln [\text{Ag}^+_1] - \frac{RT}{nF} \ln [\text{Ag}^+_2]$$

If the cell EMF is plotted against the natural log of the changing concentration ($[\text{Ag}^+_1]$), the slope will be found to be RT/nF and the intercept will have the value of $-\frac{RT}{nF} \ln [\text{Ag}^+_2]$. The intercept, therefore, when divided by the slope will give the value of $-\ln [\text{Ag}^+_2]$, from which the value of K_{sp} for silver chloride can easily be calculated.

Procedure:-

Switch on the computer and select the LoggerPro application. Load the file which sets up the data logging process for this experiment. Insert the voltage probe into the interface and click on the Collect button to display the voltage.

Prepare two silver electrodes by dipping them in 1 mol dm^{-3} nitric acid for 10 – 20 seconds and then rinse with distilled water. Store the electrodes in distilled water until required.

Prepare a saturated solution of silver chloride by adding 1 cm^3 of 0.05 mol dm^{-3} silver nitrate solution to 50 cm^3 of distilled water, followed by 1 cm^3 of 0.05 mol dm^{-3} potassium

chloride solution. The volumes must be accurately dispensed and the silver chloride solution stored in a dark cupboard until required.

Prepare a series of silver nitrate solutions by diluting the standard 0.1 mol dm^{-3} solution according to the following table:-

Volume of water/cm ³	Volume of silver nitrate/cm ³	[Ag ⁺]/mol dm ⁻³
50	1.0	1.96×10^{-3}
50	2.0	3.85×10^{-3}
50	5.0	9.09×10^{-3}
25	5.0	1.67×10^{-2}
25	10.0	2.86×10^{-2}
0.0	25.0	1.00×10^{-1}

Place 25 cm^3 of each solution in separate clean, dry 50 cm^3 beakers. Prepare salt bridges for each beaker by soaking strips of filter paper in 1 mol dm^{-3} potassium nitrate solution. Leave the strips in the potassium nitrate solution until they are required.

Place 25 cm^3 of the silver chloride solution in a small, dry beaker and dip one of the silver electrodes into the solution. Place the beaker containing the most concentrated silver nitrate solution next to the silver chloride solution and dip the second silver electrode into the silver nitrate solution. Fold one of the filter paper strips and dip the ends into the solutions to form a salt bridge between the solutions. Connect the electrodes to the voltage probe - if the computer displays a negative reading, change round the connections to the electrodes.

When the voltage reading is steady, click on the Keep button and enter the concentration of the solution when the prompt window appears. Take out the electrode from the silver nitrate solution, rinse it with distilled water and dry it with a paper tissue. Place the electrode in the next solution and use a new salt bridge to complete the cell. Record the new voltage when the reading is steady and enter the concentration of the solution. Repeat this procedure for each solution and finally click on the Stop button to end the data logging part of the experiment.

Return all the silver solutions to the silver residues bottle (do NOT pour them into the sink!).

Data Processing:-

Plot a graph to show –

- the relation between cell EMF and silver nitrate concentration
- the relation between cell EMF and the natural log of the silver nitrate concentration.

From the Nernst equation given above, the slope of the second graph will give the value of RT/nF and the intercept will give the value of $-RT/nF \ln [\text{Ag}^+_{\text{c2}}]$. Dividing the intercept by the slope therefore gives $-\ln [\text{Ag}^+_{\text{c2}}]$. Taking the antilog_e gives the concentration of silver ions in the saturated solution of silver chloride.

Since the solution contains equal concentrations of Ag^+ and Cl^- ions -

$$\begin{aligned} & [\text{Ag}^+] = [\text{Cl}^-] \\ \text{and therefore} & \\ & K_{\text{sp}} = [\text{Ag}^+]^2 \end{aligned}$$

This experiment can be repeated using silver bromide (prepared from silver nitrate and potassium bromide) or silver iodide (prepared from silver nitrate and potassium iodide) to demonstrate the wide range of concentrations to which the Nernst equation can be applied.

Analysis of the Hydrogen Emission Spectrum

INTRODUCTION:-

The emission spectrum of hydrogen has been studied for over 120 years. Quite early on, a formula was discovered which showed that the four lines in the visible spectrum belonged to a series. The Balmer equation (1885) in modern symbols is –

$$s = R \left(\frac{1}{2^2} - \frac{1}{n^2} \right)$$

where σ = wave number (no. of cycles per metre)

R = Rydberg's constant

n = an integer from 3..... onwards.

Bohr interpreted this equation as indicating that each line in the spectrum corresponded to the emission of the energy difference between two electronic energy levels, leading to the conclusion that energy levels were quantised.

PROCEDURE:-

Launch the LoggerPro software application and from the Sample Data folder, select Physics, then Gas Discharge Spectra and finally Hydrogen. Select Examine from the Analysis menu and identify as accurately as possible the wavelength of each of the four wavelengths – the third and fourth peaks are very small but they can be identified, with care. A fifth peak at approximately 389 nm can be located under favourable conditions.

DATA ANALYSIS:-

Construct a spreadsheet in which the experimental data are placed in columns headed n (for quantum number) and wavelength/nm. The first column is the quantum number column and should contain the integers from 3 to 6 (or 7 if you have located the fifth peak). The second column should be titled $1/n^2$. The third column is the wavelength column, with the data recorded in nm. The fourth column is the wave-number column and should contain the formula $=1 \times 10^9/x$ where x refers to the cell containing the wavelength.. This formula is then to be copied for all the wavelength data.

Plot wave-number against $1/n^2$ and determine the best linear fit, selecting the option to display the regression equation on the graph. The intercept gives the value of the Rydberg constant (literature value – $1.097 \times 10^7 \text{ m}^{-1}$).

The Rusting of Iron – Rate of Reaction

INTRODUCTION:-

The rusting of iron is a common example of a heterogeneous reaction (ie the reactants are present in different phases or states). The surface of a solid is never perfectly uniform and chemical reactions tend to take place at active sites on the surface. The rate of reaction depends on the number of active sites on the surface and the concentration or pressure of the reactant in the other phase. The study of the rates of reactions at surfaces is important for a number of catalysed reactions, including those involving enzymes.

In this experiment, steel wool is cleaned by dipping it in propanone or ethanol to remove grease and then left to oxidise.

PROCEDURE:-

Launch the LoggerPro software and connect the pressure sensor to the Labpro interface. Weigh approximately 1 g of steel wool and immerse it for 30 seconds in a 1 mol dm⁻³ solution of ethanoic acid. Remove the steel wool with forceps or tongs and rinse it in distilled water and then either propanone or ethanol. Dry the steel wool between two sheets of filter paper and then tease it apart before inserting it in a 100 cm³ conical flask. Stopper the flask firmly with the pressure sensor unit and click on the **Collect** button.

Leave the sensor to collect data for a period of 25 – 30 minutes and then click on the **Stop** button to halt the data collection. From the **Data** drop down menu select **Store Latest Run**. Select **Data Collection** from the **Experiment** menu and change the number of readings per minute to 4. Remove the pressure sensor unit from the flask.

While the experiment is proceeding, prepare a water bath large enough to contain another 100 cm³ conical flask by adding 300 cm³ of water to a 500 cm³ beaker. Heat the beaker and its contents to 40⁰ C and clamp another 100 cm³ conical flask in the water bath. Weigh another 1 g of steel wool and clean it as before. Place it in the flask and insert the pressure sensor unit from the flask at room temperature. Click on the **Collect** button to begin collecting data and leave the sensor to collect data for 10 – 15 minutes. Click on the **Stop** button and then refer to the on-screen help for the data processing procedure.

INTERPRETATION:-

The data will show whether the rate of reaction is determined by the pressure of oxygen alone or whether the rate is determined by both the pressure of oxygen and the number of active sites on the surface of the metal.

As the reaction proceeds, the active sites which have reacted with oxygen are inactivated because the product of the reaction does not move away. What are you able to say about the number of active sites on this sample of steel wool?

Moving On From Enthalpy of Neutralisation

One of the simplest and most successful of data logging experiments is the measurement of the temperature change on mixing acid and alkaline solutions. The same procedure can be applied to a number of other exothermic reactions, particularly if enquiry based experiments are required.

The following suggestions are offered– there are, however, many internet sites which offer a choice of procedures and greater detail:-

Copper sulphate solution plus excess concentrated ammonia solution, to measure the displacement of water by ammonia to give the $\text{Cu}(\text{NH}_3)_4^{2+}$ ion. Compare this with the reaction of copper sulphate solution with excess ethylenediamine solution.

To a solution of copper sulphate, add the same quantity (ie the same number of moles) of ethylenediamine, measure the temperature rise, then add a further quantity of the diamine and measure the temperature rise and, finally, a third quantity of the diamine. Repeat the experiment with a solution of a nickel salt (eg nickel sulphate) and comment on the difference.

A recommended reference:-

<http://www.chemtopics.com/aplab/complexions.pdf>

Since the quantity of heat evolved in an exothermic reaction is directly proportional to the amount of reactant(s), it ought to be possible to employ titration techniques to determine the concentration of a given reactant solution. This doesn't usually work because solutions have to be quite concentrated if they are to give measurable temperature changes and small errors in measuring volumes can lead to large errors in calculating concentrations. This kind of titration is, however, quite useful in determining the stoichiometry (ie the ratios in which reactants combine) of a reaction.

For example, a temperature sensor is placed in a measured volume (eg 25 cm^3) of reactant 1, of known concentration. The data collection is set for data entry from the computer. Display the temperature in a small digital window. A small volume (eg 5 cm^3) of reactant 2 is added and the mixture stirred, without stopping the data logging. As soon as the temperature begins to level off, keep the temperature reading and enter the volume of reactant 2 in the pop-up window. Add the second portion of the reactant 2 solution wait for the levelling off and record the temperature value. This process is repeated until an appreciable excess of reactant 2 has been added. The graph should show a rising linear portion followed by an almost horizontal portion. Where these intersect is the volume of reactant 2 which reacts completely with reactant 1. From this information the reacting ratio of the two reactants can be calculated.

Sources for Investigative Assignments

A common criticism expressed by Board examiners is that schools offer only an extremely limited range of systems for investigation. Since its inception in 1963, **Education in Chemistry** has always featured articles of a practical nature. Most of these are still useful in A Level courses and only those relating to the use of computers for data logging now need significant updating.

Since 1987, every school in which chemistry is taught, has received a free copy of the journal and teachers may find it useful to have a brief review of the major articles that may serve as a jumping-off point for investigative assignments..

Design for an experiment, I Williams & R G Hacker, 7, 20 – 24 (1970)

This is an important article, warning that even very basic chemical reactions are seldom as simple as they may, at first, appear. It should be read by all teachers supervising assignments. Another paper, dealing specifically with the rate of dissolution of calcium carbonate in hydrochloric acid occurs in H Bassow et al, *J Chem Ed*, 48, 327 – 329 (1971).

Comparative Hydrolysis of Tertiary Butyl Halides, Pritchard, 15, 91 – 92 (1978)

An experimental procedure is described and sample data provided.

Simple Kinetic Experiments for Schools, J P Lorimer & T J Mason, 21, 161 – 162 (1984) and 22, 19 – 21, (1985)

Investigation of SN1 and SN2 hydrolysis of chloro-, bromo- and iodo-alkanes.

Solvent Choice for Alkyl Halide Hydrolysis, J P Lorimer, T J Mason & P H Bull, 22, 110 – 111 (1985)

Similar in content to the above.

Bromine/Formic Acid Reaction Revisited, P M Osborne & B Frere, 23, 14 – 15 (1986)

Although this is primarily a data logging experiment using the BBC Computer, there is sufficient chemical detail for reaction mixtures to be prepared for use with other data loggers or manual recording of data.

Potentiometry and Kinetics, B A Ardron, J G Dawber & E G Taylor, 23, 151 – 152 (1986)

Two reactions – bromine and methanoic acid, peroxodisulphate and iodide – are considered. The emphasis is on the underlying theory of how the Nernst equation may be applied but there is enough practical detail to allow an investigation to be planned.

A Light Intensity Comparator, P F Knewstubb, 25, 17 – 19, (1988)

Most of this article deals with the construction of a comparator but there are some experimental details for the investigation of the reaction between bromine and cinnamic acid the reaction between iodide and acidified bromate.

Teaching Practical Kinetics, O El Seoud, M El Seoud, P A R Pires and K Takashima, 34, 22 – 23, (1997)

Three methods – pH, colorimetric and conductimetric – are suggested for the monitoring of the hydration of ethanoic anhydride to ethanoic acid. Sample data are also provided.

Saponification Kinetics Revisited, J G Wheeler & W E L Cockcroft, 34, 47 – 48, (1997)

The reaction between sodium hydroxide and ethyl ethanoate is described using concentrations which give quite a rapid reaction rate and following the reaction by conductivity measurements. Sample data are given and the procedure could easily be adapted for data logging.

Saponification in a Syringe, J Hoppé, 35, 21 –22, (1998)

The second order kinetics of the saponification reaction are studied, using the sodium/potassium salts of acid esters.

Inorganic Reaction Mechanisms – Dispelling a Myth, J F Packer, 35, 75 – 76, 79 (1998)

Although the experimental details are rather sparse, there is an interesting discussion of inorganic mechanisms in connection with the reactions between thiosulphate and iodine, peroxodisulphate and iodide and iron catalysed reactions.

The American Chemical Society's education journal (J Chem Ed) is an invaluable source of material which can be adapted for use at Advanced Level. Recent articles include –

Spontaneous Hydrolysis of Methyl Chloroformate, O E El Soud, K Takashima, 75, 1625 – 1627 (1998)

Pseudo-first order reaction, moderately fast reaction (30 - 45 min half-life), may be followed by pH, conductivity and colorimetric measurements.

Nernst Equation: Determination of Equilibrium Constants, M L Thompson, L J Kateley, 76, 95 – 96 (1999)

Basic use of Nernst equation to measure $[Ag^+]$ in silver nitrate/ammonia mixtures, could be applied to determination of copper complexes if silver electrodes are not available.

Investigating Aspects of Catalase Activity, D R Kimbrough, MA Magoun, M Langfur, 74, 210 - 212 (1997)

Hydrogen peroxide is used as substrate and catalase is extracted from a large number of plant sources, liver and milk. The method adopted is based on UV absorption but pressure measurement would be more practicable in schools.

Methylene Blue – Ascorbic Acid, T Snehalatha, K C Rajanna, P K Salprakash, 74, 228 – 233 (1997)

Colorimetric logging of reaction rate, with extensive sample data and analysis of results.

Kinetics Experiment to Demonstrate the Role of a Catalyst, C L Copper, E Koubek, 75, 87 – 89 (1998)

Iodide ion as a catalyst for the reaction with hydrogen peroxide, described as a clock reaction but could be modified for data logging with a colorimeter.

Internet sources have been, for the most part, confined to a few reactions – thiosulphate and hydrochloric acid and the iodine clock. The Regensburg University web-site, however offers a range of different experiments, including ester saponification and hydrolysis. The procedures for the SN1 and SN2 hydrolysis of alkyl halides and enzyme kinetics may easily be modified for use with data logging systems available in Britain. The texts are in English and the translation falters only occasionally, but compounds are usually referred to by “traditional” names rather than the IUPAC ones. The experiments come with varying amounts of background theory, an outline procedure and extensive sample data and spreadsheet illustrations – the index page to this very useful collection is to be found at – www.uni-regensburg.de/Fakultaeten/nat_Fak_IV/Organische_Chemie/Didaktik/Keusch/indexe.html. (NB There is an underline character between Organische_ Chemie.)

The kinetics of oxidation of bromocresol green by sodium hypochlorite is presented at www.nsm.smcm.edu/Chemistry/chem-gchemlab/WebLab/ExptSplit/LAB14-1TR.htm with an extensive background discussion.

The behaviour of catalase, extracted from turnip, horseradish or potato, is investigated at www.mhhe.com/biosci/genbio/dolphin52/topic5.html. A detailed description of the preparation of the reagents is given but the experimental procedure is limited to a few suggestions and no sample data are given. Whilst this might be frustrating for a teacher looking for an experimental procedure to demonstrate the characteristics of enzyme kinetics, it is on the other hand, an excellent starting point for a student investigation.

The substitution reaction involving iodine and propanone appears on many web pages but www.dartmouth.edu/~chemlab/chem6/kinet1/full_text/procedure.html proposes the use of cyclohexanone as an alternative ketone. It is important to remember that plastic cuvettes may not be inert to solutions containing ketones and may also become stained if iodine solutions are left in them for more than a few minutes.

An interesting investigation into water quality monitoring is described on the web-site departments.oxy.edu/tops/Waterquality/waterstudent1.htm in which baker’s yeast and diluted milk are used as a model for the bio-remediation of polluted waters. This web-page comprises part III of the experiment, giving an introduction and a comprehensive procedure, together with some questions to be answered on the basis of the results obtained.

A clock procedure is used to study relative rates of hydrolysis of alkyl halides on web.uccs.edu/bgaddis/leadership/expt9_1D4.htm. There are no sample data but the experimental directions are clear and there is an excellent summary of the relevant background, together with a post-lab assignment of nine questions to be answered on the basis of the results obtained.

A useful manual of experiments involving the use of a simple fuel cell is located at www.ectechinc.co.uk/exps.html - experimental details are very basic but there are useful references to other internet sites.

A purely theoretical web-site at server.chem.ufl.edu/~itl/2041/lec_m.html has some very attractive graphics to illustrate the temperature dependence of reaction rates. Further useful guides occur at www.chemguide.co.uk/mechmenu.html - this is the menu page for some excellent material on organic mechanisms pitched at GCE A Level pupils – and www.ksw.org.uk/departments/chemistry/sn2.html has a useful summary of Nucleophilic Substitution.

Internet sites were accessed during June 2003.

The RSC Library at Burlington House holds complete runs of Education in Chemistry and the Journal of Chemical Education, from their inception. The other major sources of material for assignments, available at the Library, are the four volumes of Demonstrations for Teachers of Chemistry, by Bassam Shakashiri (useful for the comprehensive references to the original literature) and the complete run of the Journal of Chemical Education published by the American Chemical Society.

The Society is anxious to improve its services to chemistry teachers and details of access to the Library resources and its photocopying charges are available from the Librarian (email - library.rsc.org, telephone 020 7437 8656, mail – LIRC, Burlington House, Piccadilly, London W1J 0BA).