

Appendix 1

Appendix 1

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| 6f, 6g, 6i | 1. Electrochemical cells | | |
| | <ul style="list-style-type: none"> Set up an electrochemical cell Read a voltmeter Record observations from test tube experiments Compare results from different types of experiment Write cell descriptions and calculate E_{cell} | <p>Electrochemical cells are constructed and their e.m.f. values measured; these results are compared with the equivalent test-tube reactions. The effect on the cell e.m.f. of varying solution concentration is investigated. The outcome of a redox reaction is analysed and the feasibility of a reaction is deduced.</p> <p>Timing: 1–1.5 hours</p> | <p>Appendix 2 A similar exercise is to be found in <i>Chemistry in Context</i> – third edition; Hill & Holman</p> <p>Practical 11</p> |
| 6g | 2. Redox experiments | | |
| | <ul style="list-style-type: none"> Record colour changes accurately Decide when to use a control experiment Design tests to assess relative oxidising ability Analyse a set of tabulated results and draw conclusions Tabulate a set of conclusions Write overall ionic equations from redox half equations Deduce order of oxidising ability and the feasibility of reactions | <p>The exercise is based on a series of test-tube reactions designed to investigate the reactions between various oxidising agents and reducing agents. Careful observations allow deductions to be made regarding the oxidising strengths of a number of different species. Ionic equations are written based on the redox half-equations provided. The feasibility of a reaction is deduced from the provided Standard Electrode Potential data.</p> <p>Note: The layout used in this exercise could be adapted for use with other reactants.</p> <p>Timing: 1 hour</p> | <p>Appendix 2</p> |

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| 6k, 6m, 6n | <p>3. Determining the value of the Avogadro constant by electrolysis</p> <ul style="list-style-type: none"> • Consolidate manipulative skills • Make precise measurements of time, current and mass • Analyse data numerically • Compare outcome with data book value and hence determine experimental error • Evaluate experimental procedures and identify potential sources of error • Suggest procedural improvements to reduce/eliminate errors | <p>The value of the Avogadro constant, A_N, may be determined using an electrolytic method. The apparatus needed, and the general approach is very similar to that used in Experiment 4. You may use your 'usual' electrolysis apparatus, or you may choose to place the two electrodes in separate beakers, joined by a salt bridge made from a glass tube filled with saturated potassium nitrate solution, as in an electrochemical cell. The latter option prevents Cu^{2+} ions from migrating to the cathode, where they would interfere with the release of hydrogen gas, and so reduce the accuracy of its volume measurement. In both cases, the electrolyte used is 0.50 mol dm^{-3} sulphuric acid.</p> <p>The electrodes are cleaned by briefly dipping (for 2–3 s) into $6 \text{ mol dm}^{-3} \text{ HNO}_3$, (care – corrosive) followed by washing (as in Exercise experiment 4). Very precise measurement is needed, so weighing the anode to 4 d.p., or a similarly precise measurement of the volume of hydrogen produced, would be best. The total charge (Coulombs) required to release one mole of copper, or hydrogen, is then calculated. From this, the number of copper atoms in one mole may be deduced (see example below). Evaluate procedure by comparison of result with data book value.</p> | Modified Experiment 4 |

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| | | <p>Calculation</p> <p>Total charge = current (A) × time (s) / Coulombs <i>e.g.</i> = $0.601 \times 1802 = 1083 \text{ Coulombs}$</p> <p>$N^\ominus e^-$ = total charge ÷ charge per e^- <i>e.g.</i> = $1083 \div 1.6022 \times 10^{-19} = 6.759 \times 10^{21} e^-$</p> <p>$N^\ominus \text{Cu}^{2+}$ = $N^\ominus e^- \div 2$ <i>e.g.</i> = $6.759 \times 10^{21} \div 2 = 3.380 \times 10^{21} (\text{Cu}^{2+})$</p> <p>Mass Cu^{2+} ions formed = mass Cu lost <i>e.g.</i> = 0.3554 g</p> <p>Moles Cu^{2+} = mass $\text{Cu}^{2+} \div A_r(\text{Cu})$ <i>e.g.</i> = $0.3554 \div 63.546 = 5.593 \times 10^{-3} \text{ mol}$</p> <p>$N^\ominus \text{Cu}^{2+}/\text{mol}$ = $N^\ominus \text{Cu}^{2+} \text{ ions} \div \text{moles } \text{Cu}^{2+} \text{ ions}$ <i>e.g.</i> A_N = $3.380 \times 10^{21} \div 5.593 \times 10^{-3}$ = 6.044×10^{23}</p> <p>The Avogadro constant = 6.04×10^{23}</p> <p>% error = $\frac{6.04 \times 10^{23} - 6.02 \times 10^{23}}{6.02 \times 10^{23}} \times 100$ = 0.332%</p> <p>Timing: 1 hour</p> | |

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| 6m | <p>4. Quantitative electrolysis</p> <ul style="list-style-type: none"> • Consolidate manipulative skills • Make measurements of time, current and mass • Analyse data numerically • Compare outcome with data book value and hence determine experimental error • Evaluate experimental procedures and identify potential sources of error • Suggest procedural improvements to reduce/eliminate errors | <p>Set up the circuit as for an electrolysis experiment, using a 6 V d.c. supply, a milliammeter (range 0–1 A), a variable resistor, two copper electrodes and an electrolysis cell (containing 0.10 mol dm⁻³ aqueous copper(II) sulphate). (Harmful, Harmful to the environment)</p> <p>Clean the electrodes with emery paper, wash the anode with water and then with methanol, (Toxic, Highly flammable) dry and weigh it accurately (to at least 3 d.p.). Partially immerse the electrodes in the copper sulphate solution and pass a current of about 0.4 A for about 30 min. Measure both the time and the current with precision. Remove the anode, wash it with water and then with methanol, dry it and reweigh it.</p> <ol style="list-style-type: none"> 1. From the loss in mass of the anode, deduce the mass and moles of copper lost. 2. From the current and time, calculate the charge which flowed though the circuit using: charge (Coulombs) = current (A) × time (s). <p>Using the answers to 1 and 2, calculate the number of Coulombs needed to remove one mole of copper.</p> <p>A charge of 193000 (2 × 96500) Coulombs is required to remove one mole of copper. The difference between this and the value above is a measure of experimental error. Calculate this error as a percentage of the accurate value (193000 Coulombs). Sources of error should be identified and suggestions made to limit/eradicate them.</p> <p>Timing: 1 hour</p> | <p><i>Classic Chemistry Experiments</i>, The Royal Society of Chemistry – Experiment 81</p> |

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| 7j, 7k | <p>5. Determination of the dissociation constant for a weak acid without pH meter</p> <ul style="list-style-type: none"> • Use a pipette and a burette to produce solutions of different concentration • Prepare a buffer solution • Use and understand indicator theory • Calculate dissociation constants | <p>This experiment allows the colour of a bromocresol solution at different pH values to be investigated by viewing the combined colour of two solutions, one solution having the [HIn] at a given pH and the other solution having the [In⁻] present at that pH. A number of combinations of solutions, covering a range of pH values, are prepared and the pH of a solution of a weak acid (containing bromocresol) is to be established by colour comparison. From this, the K_a value of the acid may be calculated</p> <p>Timing: 1 hour</p> <p>Note: More interesting than using universal indicator. Explores indicator theory.</p> | <p>Appendix 2</p> |

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| 7j, 7k, 7l, 7m | <p>6. Measuring the K_a value of a weak acid (PLANNING)</p> <ul style="list-style-type: none"> Plan a quantitative exercise, considering approach, quantities, precision, apparatus and data analysis Calculate quantities needed to make a buffer solution Devise a system to test the effectiveness of the buffer solution <p>Suggested assessment points</p> <ol style="list-style-type: none"> Setting up and buffering pH meter/electrode Titration: Rinsing pipette and burette; HA in pipette; measures volumes NaOH and pH; records volume – smaller intervals near pH 7; continues adding excess NaOH. pH curve: axis/scales/shape correct, fits scales, shows working to get pK_a from $V/2$. Buffer: quotes volumes NaOH/HA to use, volume NaOH = $V/2$; adds small volume of named strong acid/base to test buffer; uses water as control; measures pH before/after each addition; compares pH change of buffer/control | <p>In this exercise students plan experiments to find the pK_a value of a weak acid, to prepare a buffer solution using that acid and to test the buffering capacity of their buffer solution.</p> <p>Background Information</p> <ul style="list-style-type: none"> The weak acid is monobasic and may be represented as HA. When a weak acid is titrated with a strong base then at the half-neutralisation point $pK_a = \text{pH}$ (the pH when half the volume of base required to exactly neutralise the acid has been added to the acid). Start with exactly $0.100 \text{ mol dm}^{-3}$ aqueous sodium hydroxide (Corrosive) and approximately $0.100 \text{ mol dm}^{-3}$ aqueous HA. A buffer solution resists large changes in pH when small amounts of acid or base are added. <p>Plan</p> <p>The plans should include the following-</p> <ol style="list-style-type: none"> Full practical detail of the experiments, including the apparatus they would use, and the measurements they would make, from which they could draw a pH curve for the reaction. A sketch of the expected pH curve, which clearly shows how the values of the neutralisation volume, $V \text{ cm}^3$, and the pK_a values are obtained. A description of how they would prepare a buffer solution by mixing accurately measured volumes of HA and sodium hydroxide. The buffer solution should have a pH equal to the pK_a value of HA. | <p>Details of similar experiments can be found in many A level practical books.</p> |

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| | | <p>Explanation of choice of volumes by making reference to the expressions:</p> $K_a = \frac{[H^+][A^-]}{[HA]} \quad pK_a = pH \text{ (at half-neutralisation point)}$ <p>4. A description of the experiments they will carry out to check the buffering capacity of the solution you have prepared. You must compare the changes in pH of your buffer with that of a control.</p> <p>Time: 1 hour Note: Some suggestions for assessment points are given but they may need to be amended or adjusted depending on the experience of the class.</p> | |

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| 7j | <p>7. Finding K_a for two weak acids</p> <ul style="list-style-type: none"> • Use a pipette and a burette with confidence and precision • Calibrate and use a pH meter • Deduce the K_a value of a weak acid from concentration and pH data • Deduce the K_a of a weak acid by graphical means • Evaluate and suggest improvements to the experiments used in the exercise | <p>In this exercise, students determine the K_a values of two different weak acids. It is not critical which weak acids are used but 0.100 mol dm⁻³ solutions of ethanoic acid (Corrosive) and chloroethanoic acid (Toxic, Harmful to the environment) work well. The aqueous sodium hydroxide used in the experiments should have an accurately known concentration of about 0.100 mol dm⁻³.</p> <p>The K_a value of the first acid (HA1) is deduced, in Experiment 1, by first determining the pH and the concentration of the weak acid solution.</p> <p>The K_a value of the second acid (HA2) is deduced, in Experiment 2, by reading the pH value at the <i>half-neutralisation</i> (half-equivalence) point from a pH graph.</p> <p>Experiment 1 Procedure</p> <ol style="list-style-type: none"> 1. Set up, buffer and calibrate a pH meter and use it to measure the pH of the weak acid solution, HA1. 2. Titrate the weak acid solution against 0.100 mol dm⁻³ aqueous sodium hydroxide using phenolphthalein as indicator. <p>Calculate the K_a value for the weak acid HA1 using the formula: $K_a = \frac{[H^+]^2}{[HA]}$</p> <p>Experiment 2 Procedure</p> <ol style="list-style-type: none"> 1. Construct a table to show the volume of aqueous sodium hydroxide added and the pH of the solution. Record the volume and pH data, obtained in the steps which follow, in this table. | <p>Details of similar experiments can be found in many A level practical books.</p> |

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| | | <ol style="list-style-type: none"> 2. Transfer 25.00 cm³ of the solution of the weak acid, HA₂, to a conical flask. Measure the pH of this solution using a pH meter. 3. From a burette, add 5.00 cm³ of aqueous sodium hydroxide. Stir the mixture and measure its pH. 4. Repeat step 3 until the neutralisation point is close. Now add the aqueous sodium hydroxide in smaller quantities as the pH changes rapidly. 5. Once the neutralisation point is passed, and the pH is no longer changing rapidly, add 5.00 portions of aqueous sodium hydroxide until a total of about 40.0 cm³ has been added. 6. Plot a graph of pH vs. volume of sodium hydroxide added. 7. From your graph read off the volume, V cm³, at the neutralisation (equivalence) point. 8. Calculate the volume of aqueous sodium hydroxide, V/2 cm³, required to half neutralise the weak acid solution. 9. The pK_a value of HA₂ is equal to the pH value at V/2 cm³. Read off this value from your graph and from it deduce the K_a value of HA₂. <p>Evaluate both experiments and suggest changes which would improve their accuracy.</p> <p>Timing: about 1 hour for each experiment</p> <p>Note: If a pH meter is not available, the pH values may be determined using Universal Indicator paper. It is strongly suggested that a series of 'narrow range' papers are used, rather than papers which are designed to cover almost the whole of the pH range.</p> | |

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| 71 | <p>8. Making a pH indicator</p> <ul style="list-style-type: none"> • Prepare extracts of a variety of coloured plant material • Test the response of these extracts to pH change • In conjunction with the buffer solutions produced in experiment 10, determine the effective range of the indicators present in your extracts | <p>A pH indicator is a substance that has a different colour when added to acid or alkali. Litmus is an extract of lichens (e.g. <i>Rocella</i>). In this experiment a pH indicator is made from red cabbage or from fresh beetroot, green cabbage or coloured flower petals such as roses or hydrangeas. A number of different extracts should be prepared and their end-point characteristics compared.</p> <p>If this experiment is performed just prior to Exercise 10 (pH Buffers and Indicators), the effective range, as well as the colour change, of each indicator may be found. The indicator extracted from red cabbage is an anthocyanin dye. A literature/internet search for the identities of the indicators present in the other natural materials used might prove to be of interest to some students.</p> <p>Procedure</p> <ol style="list-style-type: none"> 1. Boil 20–30 g of red cabbage in about 100 cm³ of tap water for about 5 min. The water should have developed a noticeable colour. 2. Allow the mixture to cool for a few minutes and filter it. 3. Place three test tubes in a rack. Half fill one with alkali, one with acid and one with deionised water. 4. To each test tube add approximately 2–3 cm³ the filtrate. 5. What colour is the indicator when neutral, when alkaline and when acidic? 6. Repeat steps 1–5 for each of the samples you have been given. <p>Timing: 1 hour</p> <p>Note: This is a fun experiment with a message. Many students are unaware of the major role played by natural products in our lives.</p> | <p><i>Classic Chemistry Experiments</i>. The Royal Society of Chemistry – Experiment 38</p> |

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| 7p, 7q, 7r | <p>9. K_{sp} and the 'common ion effect'</p> <ul style="list-style-type: none"> • Make up standard solutions and prepare equilibrium mixtures • Plot a graph • Calculate concentrations and deduce K_{sp} values • Calculate experimental errors and apparatus errors and use these to evaluate experimental accuracy • Consider the implications of apparatus errors when undertaking a planning exercise • Understand the common ion effect | <p>This experiment explores the common ion effect by measuring the changes in solubility of KIO_4 as the concentration of K^+ ions is varied. A value for $K_{sp}(KIO_4)$ is obtained. There is a detailed error analysis that compares the inherent apparatus errors when applied to large and small volumes. The exercise invites discussion of the implications of this when choosing equipment and quantities in a planning exercise.</p> <p>Timing: 1.5–2 hours</p> | <p>Appendix 2</p> |

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|------------------|--|-------|---------|----|-----|---|---|-----|-----|---|-----|----|-----|-----|-----|----|-----|----|-----|-----|-----|----|-----|----|-----|-----|-----|----|-----|----|-----|-----|-----|----|-----|----|-----|-----|-----|----|------|----|-----|-----|-----|----|------|----|-----|-----|-----|----|------|----|-----|-----|-----|----|------|----|-----|-----|-----|----|------|----|-----|-----|----|-----|--|--|--|--|--|
| 7I, 7n | <p>10. pH Buffers and Indicators</p> <ul style="list-style-type: none"> Prepare buffer solutions of known pH values. Determine the effective range and colour changes for a variety of indicators Prepare a universal indicator and investigate its colour changes over a wide pH range Deduce the composition of, and prepare, a buffer solution of given pH value Understand the buffering effect <table border="1"> <thead> <tr> <th>pH</th> <th>A</th> <th>B</th> <th>pH</th> <th>A</th> <th>B</th> </tr> </thead> <tbody> <tr><td>2.0</td><td>195</td><td>5</td><td>7.5</td><td>92</td><td>108</td></tr> <tr><td>2.5</td><td>184</td><td>16</td><td>8.0</td><td>85</td><td>115</td></tr> <tr><td>3.0</td><td>176</td><td>24</td><td>8.5</td><td>78</td><td>122</td></tr> <tr><td>3.5</td><td>166</td><td>34</td><td>9.0</td><td>69</td><td>131</td></tr> <tr><td>4.0</td><td>155</td><td>45</td><td>9.5</td><td>60</td><td>140</td></tr> <tr><td>4.5</td><td>144</td><td>56</td><td>10.0</td><td>54</td><td>146</td></tr> <tr><td>5.0</td><td>134</td><td>66</td><td>10.5</td><td>49</td><td>151</td></tr> <tr><td>5.5</td><td>126</td><td>74</td><td>11.0</td><td>44</td><td>156</td></tr> <tr><td>6.0</td><td>118</td><td>82</td><td>11.5</td><td>33</td><td>167</td></tr> <tr><td>6.5</td><td>109</td><td>91</td><td>12.0</td><td>17</td><td>183</td></tr> <tr><td>7.0</td><td>99</td><td>101</td><td></td><td></td><td></td></tr> </tbody> </table> <p style="text-align: center;">Table 1</p> | pH | A | B | pH | A | B | 2.0 | 195 | 5 | 7.5 | 92 | 108 | 2.5 | 184 | 16 | 8.0 | 85 | 115 | 3.0 | 176 | 24 | 8.5 | 78 | 122 | 3.5 | 166 | 34 | 9.0 | 69 | 131 | 4.0 | 155 | 45 | 9.5 | 60 | 140 | 4.5 | 144 | 56 | 10.0 | 54 | 146 | 5.0 | 134 | 66 | 10.5 | 49 | 151 | 5.5 | 126 | 74 | 11.0 | 44 | 156 | 6.0 | 118 | 82 | 11.5 | 33 | 167 | 6.5 | 109 | 91 | 12.0 | 17 | 183 | 7.0 | 99 | 101 | | | | <p>Buffer solutions covering a wide pH range can be prepared by mixing solutions of boric acid, citric acid (Irritant) and sodium phosphate in different proportions.</p> <ul style="list-style-type: none"> Solution A is a mixture containing 0.200 mol dm⁻³ boric acid and 0.0500 mol dm⁻³ citric acid Solution B is 0.100 mol dm⁻³ sodium phosphate (Na₃PO₄) <p>Table 1 shows the volumes needed to make 200 cm³ of a given buffer. These buffer solutions can be used to investigate the effective ranges, and colour changes, of acid–base indicators and to demonstrate the ‘buffering effect’. Table 2, which gives these data, should be used to check students’ results.</p> <p>It is unlikely that all of the indicators in Table 2 will be used, but the indicators chosen should cover as much of the pH range as possible. An individual student, or a small group could tackle the exercise but it would be just as effective, and less time-consuming, if it were undertaken as a class practical.</p> <p>A simple universal indicator (pH range of 1–13) may be prepared by dissolving thymol blue, methyl red, methyl orange, phenolphthalein and bromothymol blue in 95% ethanol (Highly Flammable). As the pH increases, the indicator changes colour from red to orange to yellow to green to blue and finally to purple.</p> <p>Students should prepare buffer solutions with pH values of 2, 3, 4 etc. and use them to investigate the colours of the individual indicators, and of the universal indicator.</p> | <p>Note: This exercise provides students with experience in preparing and using buffer solutions and indicators – both traditionally difficult topics. The chemistry involved in this buffering system is complex but the working of the universal indicator, particularly if students are able to use each of the component indicators separately, will appeal to the artist among them!</p> |
| pH | A | B | pH | A | B | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 2.0 | 195 | 5 | 7.5 | 92 | 108 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 2.5 | 184 | 16 | 8.0 | 85 | 115 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 3.0 | 176 | 24 | 8.5 | 78 | 122 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 3.5 | 166 | 34 | 9.0 | 69 | 131 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 4.0 | 155 | 45 | 9.5 | 60 | 140 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 4.5 | 144 | 56 | 10.0 | 54 | 146 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 5.0 | 134 | 66 | 10.5 | 49 | 151 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 5.5 | 126 | 74 | 11.0 | 44 | 156 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.0 | 118 | 82 | 11.5 | 33 | 167 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6.5 | 109 | 91 | 12.0 | 17 | 183 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 7.0 | 99 | 101 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

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| | <table border="1"> <thead> <tr> <th data-bbox="277 304 405 331"><i>Indicator</i></th> <th data-bbox="524 304 600 363"><i>pH range</i></th> <th data-bbox="651 304 748 363"><i>Colour change</i></th> </tr> </thead> <tbody> <tr> <td data-bbox="277 368 434 395">methyl violet</td> <td data-bbox="524 368 613 395">0.0–1.6</td> <td data-bbox="651 368 779 427">yellow to blue-violet</td> </tr> <tr> <td data-bbox="277 432 421 459">thymol blue</td> <td data-bbox="524 432 613 459">1.2–2.8</td> <td data-bbox="651 432 801 459">red to yellow</td> </tr> <tr> <td data-bbox="277 464 495 491">bromophenol blue</td> <td data-bbox="524 464 613 491">3.0–4.6</td> <td data-bbox="651 464 779 523">yellow to blue-violet</td> </tr> <tr> <td data-bbox="277 528 450 555">methyl orange</td> <td data-bbox="524 528 613 555">3.2–4.4</td> <td data-bbox="651 528 815 587">red to yellow-orange</td> </tr> <tr> <td data-bbox="277 592 405 619">methyl red</td> <td data-bbox="524 592 613 619">4.4–6.2</td> <td data-bbox="651 592 801 619">red to yellow</td> </tr> <tr> <td data-bbox="277 624 353 651">litmus</td> <td data-bbox="524 624 568 651">5–8</td> <td data-bbox="651 624 801 651">pink to blue</td> </tr> <tr> <td data-bbox="277 655 510 683">bromocresol purple</td> <td data-bbox="524 655 613 683">5.2–6.8</td> <td data-bbox="651 655 734 715">yellow to purple</td> </tr> <tr> <td data-bbox="277 719 488 746">bromophenol red</td> <td data-bbox="524 719 613 746">5.2–6.8</td> <td data-bbox="651 719 801 746">yellow to red</td> </tr> <tr> <td data-bbox="277 751 495 778">bromothymol blue</td> <td data-bbox="524 751 613 778">6.2–7.6</td> <td data-bbox="651 751 734 810">yellow to blue</td> </tr> <tr> <td data-bbox="277 815 405 842">cresol red</td> <td data-bbox="524 815 613 842">7.2–8.8</td> <td data-bbox="651 815 801 842">yellow to red</td> </tr> <tr> <td data-bbox="277 847 421 874">thymol blue</td> <td data-bbox="524 847 613 874">8.0–9.6</td> <td data-bbox="651 847 734 906">yellow to blue</td> </tr> <tr> <td data-bbox="277 911 472 938">phenolphthalein</td> <td data-bbox="524 911 636 938">8.0–10.0</td> <td data-bbox="651 911 801 970">colourless to pink</td> </tr> <tr> <td data-bbox="277 975 450 1002">alizarin yellow</td> <td data-bbox="524 975 636 1002">10.0–12.0</td> <td data-bbox="651 975 779 1034">yellow to red-violet</td> </tr> </tbody> </table> <p data-bbox="490 1023 595 1050" style="text-align: center;">Table 2</p> | <i>Indicator</i> | <i>pH range</i> | <i>Colour change</i> | methyl violet | 0.0–1.6 | yellow to blue-violet | thymol blue | 1.2–2.8 | red to yellow | bromophenol blue | 3.0–4.6 | yellow to blue-violet | methyl orange | 3.2–4.4 | red to yellow-orange | methyl red | 4.4–6.2 | red to yellow | litmus | 5–8 | pink to blue | bromocresol purple | 5.2–6.8 | yellow to purple | bromophenol red | 5.2–6.8 | yellow to red | bromothymol blue | 6.2–7.6 | yellow to blue | cresol red | 7.2–8.8 | yellow to red | thymol blue | 8.0–9.6 | yellow to blue | phenolphthalein | 8.0–10.0 | colourless to pink | alizarin yellow | 10.0–12.0 | yellow to red-violet | <p>They could then be asked to create a buffer solution having one of the intermediate pH values (3.5, 4.5, etc.); a different value for each student/group. The universal indicator could then be used to judge the accuracy of the buffers created. The buffering effect is demonstrated in point 7 below.</p> <p>Procedure</p> <ol style="list-style-type: none"> Using the volumes of solutions A and B from Table 1, prepare buffer solutions for pH 2.0 to pH 12.0. Measure their pH using a pH meter (if available). Use these buffers to investigate the effective range and the colour change of the indicators provided. Prepare a 'Universal Indicator' by dissolving methyl red (0.04 g), methyl orange (0.02 g), phenolphthalein (0.02 g), thymol blue (0.10 g), and bromothymol blue (0.08 g) in 100 cm³ of 95% ethanol. Use the buffers prepared in part 1 to investigate the colour of the UI solution across the pH range 2–12. Obtain a pH value from your teacher; deduce the volumes of A and B required and then prepare it. Use the universal indicator or a pH meter to find its pH. Add a few drops of 1.0 mol dm⁻³ sodium hydroxide to a test tube containing 3 cm³ of the pH 7 buffer, and also to a control tube containing 3 cm³ of deionised water. Add a few drops of 1.0 mol dm⁻³ hydrochloric acid (Corrosive) to a test tube containing 3 cm³ of the pH 7 buffer, and also to a control tube containing 3 cm³ of deionised water. To all four tubes, add a few drops of bromothymol blue indicator. Record and explain your results. <p>Timing: 1–1.5 hours</p> | |
| <i>Indicator</i> | <i>pH range</i> | <i>Colour change</i> | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| methyl violet | 0.0–1.6 | yellow to blue-violet | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| thymol blue | 1.2–2.8 | red to yellow | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| bromophenol blue | 3.0–4.6 | yellow to blue-violet | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| methyl orange | 3.2–4.4 | red to yellow-orange | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| methyl red | 4.4–6.2 | red to yellow | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| litmus | 5–8 | pink to blue | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| bromocresol purple | 5.2–6.8 | yellow to purple | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| bromophenol red | 5.2–6.8 | yellow to red | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| bromothymol blue | 6.2–7.6 | yellow to blue | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| cresol red | 7.2–8.8 | yellow to red | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| thymol blue | 8.0–9.6 | yellow to blue | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| phenolphthalein | 8.0–10.0 | colourless to pink | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| alizarin yellow | 10.0–12.0 | yellow to red-violet | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

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| 8e, 8k, 9.5g, 9.5n | <p>11. Catalysis – use of Fe³⁺ in I⁻/S₂O₈²⁻ reaction</p> <ul style="list-style-type: none"> Perform a 'rates' experiment with accuracy and precision Understand the use of transition metal ions as homogeneous catalysts in terms of the variability of their oxidation states | <p>The oxidation of iodide ions by peroxodisulphate ions, S₂O₈²⁻, may be represented by the equation below.</p> $2\text{I}^{-}(\text{aq}) + \text{S}_2\text{O}_8^{2-}(\text{aq}) \rightleftharpoons 2\text{SO}_4^{2-}(\text{aq}) + \text{I}_2(\text{aq})$ <p>As the reaction is between two negatively charged ions, repulsion between these ions causes it to have a high activation energy and so it is relatively slow at room temperature. Transition metals ions can act as homogeneous catalysts in this reaction; being positive, they are attracted to the appropriate negative ion. When Fe³⁺(aq) ions are present, iodide ions reduce them to Fe²⁺(aq) ions which are then oxidised back to Fe³⁺(aq) ions by S₂O₈²⁻(aq) ions.</p> <p>This exercise is essentially an 'iodine clock' reaction. A convenient way to measure the rate of this reaction is to add a fixed volume of aqueous sodium thiosulphate to the reaction mixture. This reacts with the free iodine formed in the reaction. When the sodium thiosulphate has been used up, free iodine is produced. As starch solution has also been added, a deep blue colour will be produced.</p> <p>Procedure</p> <ol style="list-style-type: none"> Mix, in a conical flask, 10 cm³ of 0.1 mol dm⁻³ aqueous sodium thiosulphate, 10 cm³ of 0.2 mol dm⁻³ aqueous potassium iodide and 5 cm³ of starch solution. Add 20 cm⁻³ of saturated aqueous potassium peroxodisulphate (Oxidising, Harmful) (about 75 g dm⁻³). Start the stop clock and swirl the flask. Note and record the time when the solution turns blue. Repeat steps 1 to 3 but, in step 1, add 0.5 cm³ of aqueous iron(III) chloride (about 0.1 mol dm⁻³). Repeat steps 1 to 3 several times, adding gradually increasing volumes of aqueous iron(III) chloride. | <p>This exercise is an extension of the 'rates' exercises described in <i>Teaching AS Chemistry Practical Skills</i>, Appendix 1.</p> |

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| | | <p>If time allows, repeat the experiment but use solutions containing $\text{Cr}^{3+}(\text{aq})$, $\text{Cu}^{2+}(\text{aq})$ or $\text{Co}^{2+}(\text{aq})$ ions in place of the aqueous iron(III) chloride.</p> <p>Timing: at least 1 hour</p> <p>Note: This exercise could be used as a test of planning skills. Students would be given the equation for the reaction and the concentration of each solution. They would then be required to write a detailed plan, including quantities and full practical details. Timing: up to 1 hour</p> | |

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| 8e, 8k, 9.5g | <p>12. Titration of $\text{MnO}_4^-/\text{H}^+$ against $\text{C}_2\text{O}_4^{2-}$ to show autocatalysis</p> <ul style="list-style-type: none"> Perform a 'rates' experiment with accuracy and precision Calculate concentrations from titration data Analyse data graphically Understand autocatalysis and 'quenching'/'stopping' a reaction | <p>Potassium manganate(VII) (Oxidising, Harmful, Dangerous to the environment) can oxidise ethanedioic acid (Harmful) as shown in the equation below.</p> $2\text{MnO}_4^-(\text{aq}) + 6\text{H}^+(\text{aq}) + 5\text{C}_2\text{O}_4^{2-}(\text{aq}) \rightarrow 2\text{Mn}^{2+}(\text{aq}) + 8\text{H}_2\text{O}(\text{l}) + 10\text{CO}_2(\text{g})$ <p>The reaction is performed without the use of a catalyst in Experiment 1 but a catalyst is added to Experiment 2. The graph for Experiment 1 will show the reaction rate increasing with time before the graph eventually moves towards a more 'normal' shape. If time permits, it might be useful for students to take gradients from this graph and to plot the rates obtained from this against time. Comparison of the graphs for the two experiments will allow the term <i>autocatalysis</i> to be introduced and explained.</p> <p>Students can investigate the catalytic action of $\text{Mn}^{2+}(\text{aq})$ ions in this reaction by repeating Experiment 1 but adding 1, 2, 4 or 6 drops of the manganese(II) sulphate solution. They will find that the addition of $\text{Mn}^{2+}(\text{aq})$ ions moves the $[\text{MnO}_4^-]$ vs. time graph to the left and also that, if sufficient $\text{Mn}^{2+}(\text{aq})$ ions are added, the increase in rate will not occur at all.</p> <p>Procedure Prepare the reaction mixtures given below using measuring cylinders. Some members of the class should do Experiment 1 and some Experiment 2 (8 steps each). The results should then be shared.</p> <p>Experiment 1</p> <ol style="list-style-type: none"> Mix 100 cm^3 of $0.200 \text{ mol dm}^{-3}$ aqueous ethanedioic acid, 5 cm^3 of 2 mol dm^{-3} sulphuric acid (Corrosive) and 95 cm^3 of water in a 500 cm^3 flask. | <p>Details of similar experiments can be found in many A level practical books.</p> |

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| | | <p>Experiment 2</p> <ol style="list-style-type: none"> Mix 100 cm³ of 0.200 mol dm⁻³ aqueous ethanedioic acid, 15 cm³ of 0.200 mol dm⁻³ aqueous manganese(II) sulphate, 5 cm³ of 2 mol dm⁻³ sulphuric acid and 95 cm³ of water in a 500 cm³ flask. Add 50 cm³ of 0.020 mol dm⁻³ aqueous potassium manganate(VII) and start timing. Shake the mixture for about half a minute to mix it well. After about a minute use a pipette and safety pipette filler to withdraw a 10 cm³ portion of the reaction mixture and run it into a conical flask. Note the time and add about 10 cm³ of 0.1 mol dm⁻³ aqueous potassium iodide. This stops the reaction and releases iodine equivalent to the residual manganate(VII) ions. Titrate the liberated iodine with 0.010 mol dm⁻³ sodium thiosulphate, adding a little starch solution near the end point. Record the titre of sodium thiosulphate. Remove further portions at least every 3 or 4 minutes. Treat each portion as described in steps 4 and 5. Continue until the titre is less than 3 cm³. Deduce, from the titre values, the concentration of MnO₄⁻ (aq) ions present in each portion. Plot a graph of [MnO₄⁻] vs. time for each experiment. <p>Compare the shapes of the two graphs. Explain any differences you see.</p> <p>Timing: 1 hour</p> <p>Note: Alternatively, the change in concentration of MnO₄⁻(aq) ions could be followed using a colorimeter. Coupling this to a computer or 'data-logger' would allow the concentration vs. time graph to be generated automatically.</p> | |

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| 8f, 11.1 | <p>13. Experiments with enzymes – the effect of enzyme concentration on rate</p> <ul style="list-style-type: none"> • Handle small volumes of liquid • Deduce organic structures • Practice mole calculations • Analyse and evaluate results | <p>This experiment requires students to investigate the effect of lipase concentration on the rate of hydrolysis of a lipid. This exercise could be used to provide practical experience prior to the students tackling the planning exercise in Experiment 14.</p> <p>Timing: 1–1.5 hours</p> | <p>Appendix 4 This experiment is based on material from the syllabus support booklet <i>Applications of Chemistry</i>.</p> |
| 8f, 11.1 | <p>14. Experiments with enzymes – planning an enzyme concentration vs. rate experiment</p> <ul style="list-style-type: none"> • Produce a detailed plan of how the experiments will be performed • Deduce the quantities of solutions to be used • Identify the apparatus needed • Decide on how the results will be analysed and evaluated • Assess the risks involved and suggest appropriate safety precautions | <p>The first part of this exercise requires students to plan an experiment to investigate the effect of an enzyme (urease) concentration on the rate of hydrolysis of urea. A fully written-up plan is provided to allow students to perform the experiment (if their own plan proves to be not feasible).</p> <p>Timing: planning – about 1 hour, experiment – about 1 hour</p> | <p>Appendix 4 This experiment is based on material from the syllabus support booklet <i>Applications of Chemistry</i>.</p> |

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| 8g, 8h, 8j | <p>15. Rate order for hydrogen peroxide/potassium iodide reaction (includes some planning)</p> <ul style="list-style-type: none"> • Measure liquid volumes using a burette • Use a stopclock • Adapt an experiment to measure the effect of a different variable • Analyse data graphically • Deduce rate orders, write a rate equation and calculate a value for the rate constant | <p>In this exercise, the effects on the initial rate of reaction of changing the concentrations of hydrogen peroxide (Corrosive), potassium iodide and acid are investigated. The data obtained are graphically analysed and the rate order with respect to each component is deduced. Full details are provided for the rate order with respect to the concentration of potassium iodide; the remaining two experiments involve some planning. Finally, students are required to evaluate their experiments and to suggest possible improvements.</p> <p>As an extension, students can be set a challenge which will develop their planning skills.</p> <p>Timing: about 1 hour per experiment. It may be better to work in groups of 3 students, with each student performing one experiment.</p> | <p>Appendix 2</p> |

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| 8g | <p>16. Rate orders for the propanone / iodine reaction</p> <ul style="list-style-type: none"> Perform a 'rates' experiment with accuracy and precision Learn about 'quenching' a reaction Calculate concentrations from titration data Analyse data graphically Deduce rate orders Write a rate equation based on the deduced rate orders Calculate the rate constant <table border="1"> <thead> <tr> <th>Mixture</th> <th colspan="5">Volume / cm³</th> </tr> <tr> <th></th> <th>1</th> <th>2</th> <th>3</th> <th>4</th> <th>5</th> </tr> </thead> <tbody> <tr> <td>I₂ solution</td> <td>30.0</td> <td>30.0</td> <td>30.0</td> <td>60.0</td> <td>30.0</td> </tr> <tr> <td>CH₃COCH₃ solution</td> <td>30.0</td> <td>60.0</td> <td>30.0</td> <td>30.0</td> <td>30.0</td> </tr> <tr> <td>HCl dilute</td> <td>30.0</td> <td>30.0</td> <td>90.0</td> <td>30.0</td> <td>60.0</td> </tr> <tr> <td>H₂O</td> <td>60.0</td> <td>30.0</td> <td>0.0</td> <td>30.0</td> <td>30.0</td> </tr> <tr> <td>Total</td> <td>150</td> <td>150</td> <td>150</td> <td>150</td> <td>150</td> </tr> <tr> <td>Initial rate / mol dm⁻³s⁻¹</td> <td></td> <td></td> <td></td> <td></td> <td></td> </tr> </tbody> </table> | Mixture | Volume / cm ³ | | | | | | 1 | 2 | 3 | 4 | 5 | I ₂ solution | 30.0 | 30.0 | 30.0 | 60.0 | 30.0 | CH ₃ COCH ₃ solution | 30.0 | 60.0 | 30.0 | 30.0 | 30.0 | HCl dilute | 30.0 | 30.0 | 90.0 | 30.0 | 60.0 | H ₂ O | 60.0 | 30.0 | 0.0 | 30.0 | 30.0 | Total | 150 | 150 | 150 | 150 | 150 | Initial rate / mol dm ⁻³ s ⁻¹ | | | | | | <p>In this exercise, students determine the rate equation for: $I_2(aq) + CH_3COCH_3(aq) \rightarrow CH_2ICOCH_3(aq) + HI(aq)$ Note: The reaction is catalysed by H⁺ ions from HCl(aq), therefore, [HCl] appears in the rate equation. The rate equation for this reaction may be written as: $R = k[CH_3COCH_3]^a[I_2]^b[HCl]^c$ Materials:</p> <ul style="list-style-type: none"> 0.0100 mol dm⁻³ I₂(aq) (2.54 g of I₂ + 8.0 g of KI in 1.0 dm³ of solution) 1.0 mol dm⁻³ CH₃COCH₃(aq) (71.0 cm³ CH₃COCH₃ in 1.0 dm³ of solution) 2.0 mol dm⁻³ HCl(aq) saturated NaHCO₃(aq) 1% starch solution distilled/deionised water <p>Assign one of the mixtures, 1–5 in the table, to each of five groups. Each group will need five 250 cm³ conical flasks, labelled A to E. In each flask, they put about 60 cm³ of saturated NaHCO₃(aq) (to quench the reaction). In a separate 250 cm³ conical flask, the assigned mixture, without the propanone (Highly Flammable), is prepared. The propanone is then added, with swirling and the clock is started. Immediately, a 25.0 cm³ portion is removed using a pipette (+ filler), and added to flask A. the time is noted when the pipette is half empty (but the clock is NOT stopped).</p> | <p>Details of similar experiments can be found in many A level practical books.</p> |
| Mixture | Volume / cm ³ | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| | 1 | 2 | 3 | 4 | 5 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| I ₂ solution | 30.0 | 30.0 | 30.0 | 60.0 | 30.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| CH ₃ COCH ₃ solution | 30.0 | 60.0 | 30.0 | 30.0 | 30.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| HCl dilute | 30.0 | 30.0 | 90.0 | 30.0 | 60.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| H ₂ O | 60.0 | 30.0 | 0.0 | 30.0 | 30.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Total | 150 | 150 | 150 | 150 | 150 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| Initial rate / mol dm ⁻³ s ⁻¹ | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

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| | | <p>Swirl flask A. this sample is now quenched and the reaction has stopped. Immediately, repeat this process by taking a second 25.0 cm³ sample of the reaction mixture, and adding it to flask B, again noting the time when the pipette is half empty. Repeat the process three more times, at about 1 minute intervals, adding the reaction solutions to flasks C, D and E, each time noting the time when the pipette is half empty. The mixtures in the flasks A to E are each titrated with 0.010 mol dm⁻³ Na₂S₂O₃(aq). From these titres the [I₂(aq)] value in each flask is calculated. A graph of time (in seconds) vs. [I₂(aq)] is drawn for each mixture. The gradient of the tangent at t = 0, is the initial rate for the reaction. The initial rates for each mixture are entered into the table. Students use this data to deduce values for the three rate orders; for example, the relationship between the initial rates in mixtures 1 and 2, gives rate order 'a'.</p> <p>Timing: 1 hour</p> <p>Note: Alternatively, the change in iodine concentration could be followed using a colorimeter. Coupling this to a computer or 'data-logger' would allow the concentration vs. time graph to be generated automatically.</p> | |

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| 9.5i, 9.5j, 9.5m, 9.5n | <p>17. Determination of the formula of complex ions</p> <ul style="list-style-type: none"> Work with care and precision Make clear and careful observations Record observations appropriately Plan a volumetric analysis exercise Deduce the formula of the complex ions formed <table border="1"> <thead> <tr> <th>tube</th> <th>vol. of 0.050 mol dm⁻³ CuSO₄ / cm</th> <th>vol. of 0.050 mol dm⁻³ 1,2-diaminoethane / cm</th> </tr> </thead> <tbody> <tr><td>1</td><td>0.0</td><td>12.0</td></tr> <tr><td>2</td><td>2.0</td><td>10.0</td></tr> <tr><td>3</td><td>3.0</td><td>9.0</td></tr> <tr><td>4</td><td>4.0</td><td>8.0</td></tr> <tr><td>5</td><td>6.0</td><td>6.0</td></tr> <tr><td>6</td><td>8.0</td><td>4.0</td></tr> <tr><td>7</td><td>9.0</td><td>3.0</td></tr> <tr><td>8</td><td>10.0</td><td>2.0</td></tr> <tr><td>9</td><td>12.0</td><td>0.0</td></tr> </tbody> </table> | tube | vol. of 0.050 mol dm ⁻³ CuSO ₄ / cm | vol. of 0.050 mol dm ⁻³ 1,2-diaminoethane / cm | 1 | 0.0 | 12.0 | 2 | 2.0 | 10.0 | 3 | 3.0 | 9.0 | 4 | 4.0 | 8.0 | 5 | 6.0 | 6.0 | 6 | 8.0 | 4.0 | 7 | 9.0 | 3.0 | 8 | 10.0 | 2.0 | 9 | 12.0 | 0.0 | <p>The source describes two experiments by which the formulae of complex ions may be deduced.</p> <p>Experiment 1 works best if the intensity of colour of the complex ion is measured using a colorimeter but it could be followed using the naked eye.</p> <p>Experiment 2 involves the displacement of one ligand (murexide) by a stronger ligand (EDTA²⁻) in an aqueous Ni²⁺ complex. The colour of the Ni²⁺/murexide complex is predominant and so the colour of the Ni²⁺/EDTA complex is only seen when all the murexide has been displaced. Murexide can therefore act as an indicator in Ni²⁺/EDTA²⁻ titrations.</p> <p>Experiment 1</p> <p>Copper(II) ions complex with 1,2-diaminoethane as shown in the equation below.</p> $\text{Cu}^{2+}(\text{aq}) + x\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2 \rightarrow [\text{Cu}(\text{H}_2\text{NCH}_2\text{CH}_2\text{NH}_2)_x]^{2+}$ <p>Procedure</p> <ol style="list-style-type: none"> Prepare the nine mixtures given in the table provided. Measure the absorption of each mixture using a colorimeter. Plot a graph of absorption against the volume of CuSO₄(aq) added. Identify the mixture which gives the maximum colour intensity. <p>Determine the molar proportions of Cu²⁺ and H₂NCH₂CH₂NH₂ present in this mixture and hence deduce the value of 'x' in the formula of the complex.</p> | <p>See <i>Chemistry in Context</i> – third edition; Hill & Holman Practical 13 for full details of these experiments</p> |
| tube | vol. of 0.050 mol dm ⁻³ CuSO ₄ / cm | vol. of 0.050 mol dm ⁻³ 1,2-diaminoethane / cm | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 1 | 0.0 | 12.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 2 | 2.0 | 10.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 3 | 3.0 | 9.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 4 | 4.0 | 8.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 5 | 6.0 | 6.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 6 | 8.0 | 4.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 7 | 9.0 | 3.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 8 | 10.0 | 2.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |
| 9 | 12.0 | 0.0 | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | | |

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| | | <p>Experiment 2</p> <p>Procedure</p> <ol style="list-style-type: none"> 1. Add 5 drops of aqueous murexide (0.5 g in 100 cm³ of water) to 5.0 cm³ of 0.1 mol dm⁻³ aqueous nickel(II) sulphate. (Harmful, Harmful to environment) 2. Using a teat pipette, add 0.1 mol dm⁻³ aqueous EDTA (disodium salt) slowly until no further changes occur. 3. Write a plan for a titration to determine the volume of aqueous EDTA with a known volume of the nickel(II) sulphate solution. 4. Discuss your plan with your teacher and then perform the titration. 5. Deduce the molar proportions of Ni²⁺ and EDTA²⁻ present at the end-point of your titration and hence deduce the formula of the Ni²⁺/EDTA²⁻ complex. <p>Timing: 1 hour per experiment</p> | |

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| 9.5j | <p>18. Ligand Exchange (Cu²⁺ complexes)</p> <ul style="list-style-type: none"> • Work with care and precision • Make clear and careful observations • Record observations appropriately • Deduce the identity and structures of the complex ions formed • Write equations for any reactions occurring | <p>In this exercise, students investigate the changes which occur when water is replaced as the ligand in the [Cu(H₂O)₆]²⁺ complex ion. Deprotonation is demonstrated by the use of hydroxide ions, and ligand replacement by the use of ammonia and chloride ions. The reversible nature of many ligand exchange processes is shown by the hydrochloric acid reaction.</p> <p>Use a stock solution of copper(II) sulphate (Harmful, Harmful to environment) (about 0.25 mol dm⁻³).</p> <ol style="list-style-type: none"> To separate samples of CuSO₄(aq): <ol style="list-style-type: none"> add dilute NaOH(aq) drop by drop, with shaking until no further changes occur add dilute NH₃(aq) drop by drop, with shaking until no further changes occur add concentrated hydrochloric acid (Corrosive) drop by drop, with shaking until no further changes occur. Then add water gradually, again until no further change is observed Describe your observations and write equations for any reactions occurring. Draw the structures, and give the name, of each complex ion present in your equations. <p>Note: The [Cu(NH₃)₄(H₂O)₂]²⁺ ion is octahedral in shape but the H₂O ligands are further away than the NH₃ ligands from the central copper ion and they are on opposite sides of the complex.</p> <p>Timing: 30 minutes</p> | <p><i>Chemistry in Context</i> – third edition, Hill & Holman Practical 23</p> <p>This covers a wide range of copper chemistry, of which this exercise is but a small part</p> |

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| 10.2j, 10.2k, 10.4d, 10.7(a) | <p>19. The preparation and purification of methyl 3-nitrobenzoate (plus possible reduction to the aryl amine)</p> <ul style="list-style-type: none"> • Determine the weight of a material using the weighing by difference method • Handle hazardous materials safely • Purify by recrystallisation • Determine the yield and melting point of a reaction product • Estimate the purity of a reaction product • Write a mechanism for the electrophilic substitution reaction involved in nitration | <p>This experiment is a fairly straightforward organic synthesis; however, the reagents used must be handled with care. The nitrated product is purified by recrystallisation and its purity is estimated by measuring its melting point. Overall, the exercise requires students to practice a wide range of organic preparative skills.</p> <p>Timing: 1.5–2 hours over two sessions</p> <p>Note: The extension of the exercise to reduce the nitro product to an aryl amine requires considerable practical experience but does offer a natural conclusion to the synthesis.</p> | <p>Appendix 2 For details of the reduction to the aryl amine see <i>Advanced Practical Chemistry</i>, J S Clarke & S Clynes (4.53) (English University Press)</p> |

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| 10.4c, 10.5e | <p>20. Triiodomethane (iodoform) test for β-hydroxy and β-keto groups</p> <ul style="list-style-type: none"> Use the 'iodoform' test to detect the presence of β-hydroxy and β-keto groups Isolate, purify and determine melting point Plan the preparation and the estimation of purity of a sample of triiodomethane | <p>This test could form a useful addition to the AS exercises on alcohols and carbonyl compounds, although it would only be assessed at A2 level. The test detects the presence of the β-keto group, $\text{CH}_3\text{CO}-$, in a compound. This group may be present either in the original compound, or because it is formed in the reaction mixture from a β-hydroxy, $\text{CH}_3\text{CH}(\text{OH})-$ group in the original compound.</p> <p>Isolating the product, recrystallising it and determining its melting point could extend the exercise. This sequence could be used as the basis of an assessment of planning skills.</p> <p>Test 1</p> <ol style="list-style-type: none"> To 0.2 cm^3 of the test sample add 2 cm^3 of 0.5 mol dm^{-3} aqueous potassium iodide and 4 cm^3 of 1.0 mol dm^{-3} aqueous sodium chlorate(I) (Oxidising, Harmful). Warm the mixture to 50 °C (323 K), using a water bath, for two minutes. Cool the mixture; fine yellow crystals separate out. <p>Note:</p> <p>Instead of using the above reagents, you may use a 4 cm^3 portion of 0.2 mol dm^{-3} aqueous iodine, to which has been added just sufficient aqueous sodium hydroxide (Corrosive) to decolourise the solution.</p> <p>Timing: 30 minutes to 1 hour</p> | <p>Details of this test can be found in many A level practical books.</p> <p>It could be combined with AS exercises 28 and 29 in Appendix 1</p> |

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| 10.5c, 10.5d, 10.5e, 11.2d, 11.2h | <p>21. Planning an experiment to identify a carbonyl compound</p> <ul style="list-style-type: none"> • Identify the relevant structural features of carbonyl compounds • Select suitable tests to use to identify an unknown carbonyl compound • Give outline details of each test, including possible observations • Outline the preparation and purification of a solid derivative • Discuss the use of spectroscopic data in confirming identity • Assess the risks involved and suggest appropriate safety precautions | <p>In this exercise, students will be assessed on their ability to plan experiments to identify a carbonyl compound. They are presented with a group of five aldehydes and ketones and they will have to use their knowledge of the chemistry of carbonyl compounds to plan the experiments. They will also be required to explain how they would use spectroscopic data to confirm their identification.</p> <p>Guidance is given regarding the approach to be adopted, and a possible mark scheme is outlined.</p> <p>Timing: up to 1 hour</p> <p>Note: This planning exercise is based on AS carbonyl chemistry, the A2 triiodomethane/iodoform test and spectroscopic analysis.</p> | <p>Appendix 2</p> |

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| 10.6e, 10.6d, 10.7k, 10.8c, 11.3c | <p>22. Nylon rope trick</p> <ul style="list-style-type: none"> Recall that polyamides are prepared by reaction between a diamine and a diacid or diol (diacid) dichloride Understand that varying the reactants alters the characteristics of a polymer Understand the relative reactivity of acid chlorides and carboxylic acids Handle hazardous materials safely | <p>The polymerisation method is termed interfacial polycondensation. This exercise produces nylon 6,10; to prepare nylon 6,6 use adipoyl chloride in place of sebacoyl chloride.</p> <p>A film of nylon forms at the interface between two immiscible liquids. When the film is lifted, it is continuously replaced, resulting in the formation of a hollow thread of nylon. This thread or 'rope' can be steadily wound onto a stirring rod, wooden spill or roller until one of the reactants is exhausted. Preparing the solutions (wear gloves and safety goggles).</p> <p>Solution A Mix 6.0 g of 1,6-diaminohexane (Corrosive, Harmful by inhalation, ingestion and skin absorption), 2.0 g of sodium hydroxide (Corrosive) and 100 cm³ of deionised water in a bottle; cork and shake to dissolve.</p> <p>Solution B Add 2.0 g (1.6 cm³) of sebacoyl chloride (Corrosive, Lachrymatory, Irritant) to 100 cm³ of hexane in a bottle; cork and shake to mix.</p> <p>Procedure</p> <ol style="list-style-type: none"> Transfer solution A to a 250 cm³ beaker and carefully pour solution B, down a glass rod, into the same beaker. Gently, using tweezers/forceps, lift the polymer film that forms at the interface and wind it onto a stirring rod/wooden spill. Wind up the nylon strand at a steady pace. | <p>A number of Internet websites show details of this reaction; some have video clips of the process.</p> <p>Also see <i>Classic Chemistry Demonstrations</i>, The Royal Society of Chemistry – Experiment 64</p> |

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| | | <p>4. Wash the polymer very thoroughly with water, ethanol or ethanol/propanone (1:1) before handling, as traces of the reactant solutions will be trapped in the polymer tube as it is lifted up.</p> <p>Timing: as a demonstration, about 10 minutes</p> | |

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| 10.6d, 10.6e, 10.7f | <p>23. Preparation of benzamide by the reaction of benzoyl chloride with ammonia</p> <ul style="list-style-type: none"> Isolate, purify and determine melting point Handle hazardous materials safely | <p>This preparation is relatively straightforward and does demonstrate just how reactive acid chlorides are. It produces a benzamide, which is a solid that is easily purified by recrystallisation. However, the reactants are very harmful and must be handled with great care. The reaction produced HCl fumes, which create pressure in the flask and so must be periodically released. Also, any benzoyl chloride spillage must be washed immediately with water. The fumes from both reactants cause severe breathing difficulties. This experiment – up to the point of recrystallisation – must be performed in a fume cupboard.</p> <p>Procedure (Use a fume cupboard)</p> <ol style="list-style-type: none"> Wear gloves. Using a measuring cylinder, transfer 25 cm^3 of '0.880' ammonia (Corrosive, Harmful to environment) and 25 cm^3 of water to a 250 cm^3 conical flask. Gradually add 10 cm^3 of benzoyl chloride (Corrosive) stopper the flask and shake well for 15 minutes (hold on to the stopper). The mixture will become warm; occasionally remove the stopper to relieve the pressure. Do not spill any of the contents. Filter off the white flakes of benzamide, wash with cold water (about 10 cm^3), and recrystallise from the minimum volume of hot water. Dry the crystals. Record your yield; determine the percentage yield, and the m.p. of the dry sample. Write a balanced equation for the reaction. <p>Timing: 1 hour (perhaps over two sessions)</p> | <p>Details of this preparation can be found in many A level practical books.</p> |

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| 10.7d, 10.7e | <p>24. Making an azo dye</p> <ul style="list-style-type: none"> • Perform a reaction under low temperature conditions • Appreciate the need to work with care and precision • Recall the chemistry of diazotisation and coupling | <p>In the exercise, an azo dye is made using simple techniques. However, great care is required in ensuring temperature control and the safe handling of materials.</p> <p>This exercise provides an attractive practical experience and coverage of the chemistry of diazotisation/coupling. It could be extended to cover: the electrophilic substitution mechanism involved, the origin of colour in extended delocalised systems, the position of substitution in a substituted arene and making other azo dyestuffs.</p> <p>Timing: 30 minutes to 1 hour</p> | <p>Appendix 4 See also <i>Chemistry in Context</i> – third edition, Hill & Holman Practical 34 and <i>Advanced Physical Chemistry</i> – J S Clarke & S Clynes (4.58)</p> |

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| 10.7f | 25. Planning the preparation and purification of <i>N</i>-phenylethanamide | | Appendix 2 |
| | Planning <ul style="list-style-type: none"> • Produce a detailed plan whereby a specified quantity of purified product may be prepared • Decide how the yield of product will be calculated • Decide how the purity of the product will be assessed • Assess the risks involved and suggest appropriate safety precautions | <p>This exercise is primarily intended to give students practice in planning an organic preparation. Their plans should cover method, apparatus, and purification of the product. The quantities proposed must take account of the percentage yield of the reaction. The plan should also include details of how the purity of the product will be estimated.</p> <p>Timing: up to 1 hour</p> <p>Note: The syllabus requires students to show an understanding of the chemistry of acyl chlorides. Given the hazards associated with this class of compound, the use of ethanoic anhydride (Corrosive) provides a safer alternate to the use of ethanoyl chloride (Flammable, Corrosive) in ethanoylation reactions.</p> | |
| 10.7f | 26. The preparation and purification of <i>N</i>-phenylethanamide | | Appendix 2 |
| | Practical <ul style="list-style-type: none"> • Determine the weight of a material using the weighing by difference method • Handle hazardous materials safely • Purify by recrystallisation • Determine yield and melting point • Estimate the purity of a product | <p>This exercise contains instructions for the preparation and purification of the product. It could be used independently, or as a follow-up experiment after the plan has been written (Experiment 25).</p> <p>Timing: 1–1.5 hours</p> <p>Note: The syllabus requires students to show an understanding of the chemistry of acyl chlorides. Given the hazards associated with this class of compound, the use of ethanoic anhydride (Corrosive) provides a safer alternate to the use of ethanoyl chloride (Flammable, Corrosive) in ethanoylation reactions.</p> | |

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| 10.8a, 10.8d, 10.8e, 10.8f, 11.3c, 11.3d | <p>27. Addition polymerisation – preparation of polystyrene / poly(phenylethene)</p> <ul style="list-style-type: none"> Handling hazardous materials safely Compare the properties of materials and suggest reasons for the differences Understand the polymerisation process | <p>Alkenes (carbon compounds containing double bonds) undergo addition reactions. In this experiment, molecules of phenylethene (styrene), the monomer, add on to each other to form poly(phenylethene) (polystyrene), the polymer.</p> <p>This experiment is only suitable for extremely able students; it may be better to demonstrate it.</p> <p>Generally, addition polymerisation is difficult to demonstrate, so it is essential to trial this experiment before showing it to the class. The quality of the product will be poor compared to commercially produced material; this could provide a useful discussion point.</p> <p>Note: Most styrene samples contain an inhibitor that must be removed by washing with 1 mol dm⁻³ sodium hydroxide solution (Corrosive), then water, in a separating funnel. The styrene is dried for 10 min. using anhydrous sodium sulphate.</p> <p>Procedure</p> <p>Wear eye protection. Work in a fume cupboard or ensure good ventilation, as styrene vapour is narcotic in high concentrations.</p> <ol style="list-style-type: none"> Add 0.1 g of di(dodecanoyl) peroxide (Irritant) to 5 cm³ of phenylethene (Highly flammable) in a boiling tube. Put a bung, containing a 20 cm length of glass tubing, in the boiling tube and clamp the boiling tube in a boiling water-bath. The bottom of this tube, which serves as an air condenser, should be positioned well away from the surface of the reaction mixture. | <p><i>Classic Chemistry Experiments</i>, The Royal Society of Chemistry – Experiment 95</p> |

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| | | <p>3. Heat for about 30 min and leave to cool. Extinguish all flames.</p> <p>4. Pour the contents of the tube into 50 cm³ of ethanol (Flammable).</p> <p>5. Use a glass rod to push the poly(phenylethene) into a lump and pour off the ethanol.</p> <p>Dry the solid on a filter paper and compare the appearance of the product with that of the starting material.</p> <p>Timing: 1 hour</p> | |

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| 10.8a, 11.3c, 11.3d | <p>28. Polymer slime</p> <ul style="list-style-type: none"> • Test chemical and physical characteristics • Revise the formation of addition polymers • Understand cross-linking by hydrogen bonding • Understand the effect that the addition of acid or alkali has on the cross-linked system | <p>This exercise involves the formation, physical and chemical testing of a cross-linked polymer. It is also good fun to do!</p> <p>Polyvinyl alcohol (PVA) is a linear polymer. Its chains contain many hydroxy (OH) groups, which make it soluble in water. Borax, $B(OH)_3$, reacts reversibly with water to form $B(OH)_4^-$, which is able to react with PVA forming cross-links between two PVA chains. Mixing solutions of PVA and borax produces a cross-linked polymeric material called 'Polymer Slime' which has some novel properties.</p> <p>Process</p> <ol style="list-style-type: none"> 1. PVA solution: Add hydrated polyvinyl alcohol (40 g) to 1 dm³ of water at 50 °C and heat, with stirring to 90 °C; or use a commercial PVA adhesive. 2. Borax solution: Add borax (40 g) to 1 dm³ of water. 3. Add 40 cm³ of PVA solution to 10 cm³ of aqueous borax. Stir vigorously until gelling is complete. <p>If wished, one drop of food colouring or fluorescein could be added to the PVA prior to mixing.</p> <p>Testing</p> <ol style="list-style-type: none"> 1. Test the slime under tension by first pulling it apart slowly, and then sharply/quickly. Also, form it into a ball and test its bouncing properties, and strike a small piece with the hand to test its response under impact. | <p><i>Classic Chemistry Experiments,</i> The Royal Society of Chemistry – Experiment 77</p> |

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| | | 2. Press a piece of the slime onto a sample of handwriting. 3. Add 0.4 mol dm^{-3} hydrochloric acid (Corrosive) dropwise, while stirring. When a change is noticed, note the number of drops added and record your observations. 4. Add 0.4 mol dm^{-3} sodium hydroxide (Corrosive) dropwise to the same sample used in 3 while stirring. When a change is noticed, record your observations. 5. Using the same sample, repeat parts 3 and 4 several times. 6. Suggest, in terms of acid/base equilibria, an explanation for the changes you observe. Timing: 1 hour Note: A fun experiment with some serious points to make about the dependence of polymeric properties on structure, on acid–base equilibria and on the pH dependence of hydrogen bonding. | |
| 10.8, 11.3c | 29. Conducting polymers <ul style="list-style-type: none"> Handle hazardous materials safely Work with delicacy and precision Measure the conductivity of a polymer Understand the polymerisation process and the concept of a conducting polymer | The exercise provides a novel way of preparing a polymer which, once formed, has unusual electrical properties. This is a relatively simple practical exercise but it does require great care and patience if it is to work properly. The chemicals used must be handled with care but the exercise is not beyond the capabilities of A level students. The polymer is formed at the anode of an electrolytic cell, with hydrogen gas being evolved at the cathode. Timing: at least 1 hour | Appendix 2 This exercise is based on an experiment found in <i>Salter's Advance Chemistry</i> . PR4 |

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| 11.1 | <p>30. Extraction of DNA from frozen peas</p> <ul style="list-style-type: none"> Extract DNA from a biological sample | <p>This is a simple but effective method of isolating DNA from peas. First the tissue is broken up mechanically. Household detergent is then used to degrade the cell and nuclear membranes, causing the membrane phospholipids and proteins to precipitate. Sodium ions from table salt cause the DNA molecules to coalesce. Heating the mixture at 60 °C partially denatures enzymes that would otherwise start to degrade the DNA into fragments. Cell fragments are separated by filtration leaving a solution containing nucleic acids and soluble protein, which is cooled in order to slow down the breakdown of DNA. A protease is then used to partially break down the soluble proteins; the nucleic acids are then precipitated into ice-cold ethanol. Keep the ethanol in a plastic bottle in a freezer overnight or stand it in an ice bath for several hours before use.</p> <p>Procedure</p> <ol style="list-style-type: none"> Dissolve 3 g of table salt in 90 cm³ of distilled water in a 250 cm³ beaker; add 10 cm³ of washing-up liquid ('watery' not concentrated type) and mix gently. Mash the peas (fresh or from frozen) using a glass rod; add the pulp to the beaker. Heat the beaker in a water-bath at 60 °C for exactly 15 minutes. Cool the mixture in an iced water bath for 5 minutes, stirring frequently, and then filter it into a second beaker; the filtrate contains DNA. Add 2–3 drops of <i>novozyme neutrase</i> (a protease) to about 10 cm³ of the pea extract in a boiling tube and mix well. | <p>The National Centre for Biotechnology Education (NCBE) publishes protocols in investigating plant DNA available from their website: http://www.ncbe.reading.ac.uk/</p> |

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| | | <p>6. Very carefully pour 10 cm³ of ice-cold ethanol (Highly flammable) down the side of the tube so that it forms a layer on top of the pea extract.</p> <p>7. Allow the tube to stand for several minutes. The DNA forms as a white precipitate in the clear alcohol layer.</p> <p>Timing: Isolating the DNA takes about 35 minutes.</p> | |
| 11.2a, 11.2c, 11.2f, 11.2g | <p>31. Separation techniques</p> <ul style="list-style-type: none"> • Use solvent extraction techniques and purify by recrystallising • Perform a titration and use a separating funnel • Determine a partition coefficient • Use and understand paper chromatography • Use and understand two-way chromatography • Use and understand electrophoresis • Understand amino acid structure and zwitterions | <p>This exercise contains a suite of four experiments designed to give practice in a range of separation techniques. The basic techniques involved are relatively straightforward but great emphasis must be placed on safety, as there are hazards associated with several of the solvents used. In some cases, the use of a fume cupboard is necessary.</p> <p>The experiments may be performed as a circus, perhaps over several weeks. If fume cupboard facilities are limited, it may be better to perform the experiments separately, so that attention can be focussed on one process at a time. The use of demonstrations might be considered; however, a hands-on approach will make the techniques more memorable to the students.</p> <p>Timing: a suite of exercises better done as a circus – allow 1 hour (at least) for each exercise.</p> | Appendix 2 |

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| Syllabus section | Skills/Learning Outcomes | Notes | Sources |
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| 11.3f, 11.3g | <p>32. Making 'Biodiesel' from rape seed oil</p> <ul style="list-style-type: none"> Compare the combustion properties of materials and their implications for large scale use Appreciate the scale of the problem of replacing conventional fuels Handle hazardous materials safely | <p>Biodiesel, a mixture of methyl esters of fatty acids, can be made very easily from a cooking oil made from rape seed, though other cooking oils may be tried. Enough biodiesel can be produced in an hour to burn, but it would not be pure enough to use in an engine. This experiment could be a starting point for further student investigations.</p> <p>A cooking oil, methanol and potassium hydroxide (a catalyst) are mixed. The resulting reaction (transesterification) produces biodiesel and glycerol as two layers. The biodiesel (top layer) is removed and washed with water to remove potassium hydroxide.</p> <p>The combustion characteristics of biodiesel can be compared with those of fossil diesel by drawing the gaseous reaction products through mineral wool (to trap particulates and test for 'sootiness') and a solution of universal indicator (to test for acidity) using a water pump.</p> <p>Procedure</p> <p>Stage 1</p> <ol style="list-style-type: none"> Weigh about 100 g of rapeseed oil into a conical flask. Carefully add 15 g of methanol (Toxic, Highly Flammable). Slowly add 1 g of a 50% (50 g per 100 cm³ of solution) potassium hydroxide solution (Corrosive). <p>Note: The chemicals can be added directly into a conical flask on a top pan balance (zero the balance after each addition).</p> <ol style="list-style-type: none"> Stir or swirl for 10 min. | <p><i>Materials</i>, The Royal Society of Chemistry – Pages 21–36 plus worksheets</p> <p>The booklet <i>Introducing Biodiesel</i> provides the background to the process</p> |

Appendix 1

| Syllabus section | Skills/Learning Outcomes | Notes | Sources |
|------------------|--------------------------|--|---------|
| | | <p>Stage 2</p> <ol style="list-style-type: none">1. Centrifuge the mixture for one minute (you will need several centrifuge tubes to deal with the quantity). If a centrifuge is not available, you should leave the mixture to settle until layers form; this will take some time.2. Decant the top layers into a boiling tube and discard the lower layers.3. Wash the product by adding 10 cm³ of distilled water to this top layer, with gentle mixing. (Do not shake the mixture).4. Repeat steps 1 and 2 once more.5. Keep your product for further investigation. <p>Timing: 1 hour</p> | |