



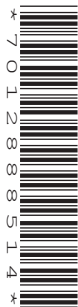
Oxford Cambridge and RSA

A Level Chemistry B (Salters)

H433/03 Practical skills in chemistry

Wednesday 20 June 2018 – Morning

Time allowed: 1 hour 30 minutes



You must have:

- the Insert (inserted)
- the Data Sheet for Chemistry B (Salters) (sent with general stationery)

You may use:

- a scientific or graphical calculator

First name										
Last name	StemUp Model Solution									
Centre number						Candidate number				

INSTRUCTIONS

- Use black ink. You may use an HB pencil for graphs and diagrams.
- The practical insert is needed with this paper.
- Complete the boxes above with your name, centre number and candidate number.
- Answer **all** the questions.
- Write your answer to each question in the space provided. If additional space is required, use the lined page(s) at the end of this booklet. The question number(s) must be clearly shown.
- Do **not** write in the barcodes.

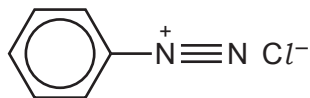
INFORMATION

- The total mark for this paper is **60**.
- The marks for each question are shown in brackets [].
- Quality of extended responses will be assessed in questions marked with an asterisk (*).
- This document consists of **16** pages.

Answer **all** the questions.

- 1 A student decides to use a microscale method to synthesise an azo dye and dye a fabric.

- (a) The student initially makes a small amount of a solution of the diazonium compound shown below, starting from an aromatic amine.



diazonium compound

Name the reagents and conditions needed to make this compound.

Reagents

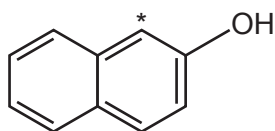
NaNO₂ (sodium nitrite) and conc. HCl, reacting with phenylamine

Conditions

below 5 °C (ice-cold solution) [3]

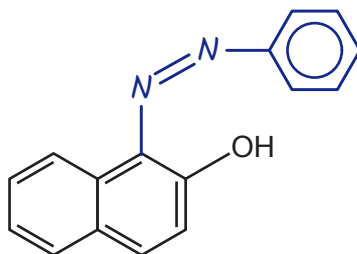
- (b) Naphthalen-2-ol, shown below, is used to make the dye. A piece of cotton is dipped into naphthalen-2-ol dissolved in sodium hydroxide. The diazonium solution is then added to dye the cotton red.

The coupling reaction involves the carbon atom marked with an asterisk, *.



naphthalen-2-ol

- (i) Complete the structure of the azo dye formed in this coupling reaction.



[1]

- (ii) The azo dye formed above has an extended delocalised system.

Describe the bonding occurring in a delocalised system and explain why this can lead to organic compounds being coloured.

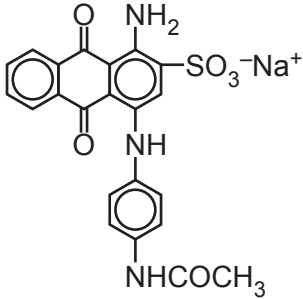
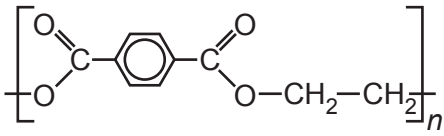
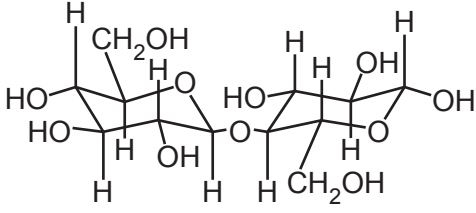
*..The azo dye has alternating double and single bonds (a conjugated system), so
 ..p-orbital electrons overlap and are delocalised over a large part of the molecule....
 ..This splits the electrons into different electronic energy levels; the larger the
 ..delocalised system, the smaller the energy gap.....
 ..The molecule absorbs visible light of a particular frequency ($\Delta E = h\nu$), which
 ..excites electrons from a lower to a higher energy level.....
 ..The remaining light that is reflected or transmitted is the complementary
 ..colour, which is the colour we see..... [4]*

- (c) Other functional groups can be attached to dye molecules and these can modify the properties of the dye.

- (i) Give **one** property of the dye that might be affected if nitro, NO_2 , groups are attached.
..the colour of the dye (it modifies the chromophore)..... [1]
- (ii) Give a **different** property affected by the attachment of sulfonate, SO_3^- , groups.
..its solubility in water: the dye becomes more soluble..... [1]

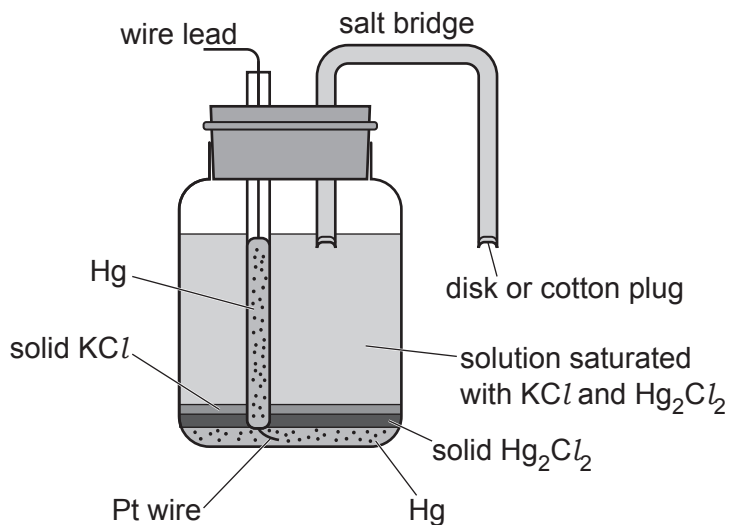
- (d) Attractions between dye molecules and polymer molecules in fabric fibres can be ionic, covalent or intermolecular bonds.

Use your knowledge of molecular interactions to fill in the empty boxes in the following table.

Type of fabric	Structure/features of polymer molecule	Structure/features of dye molecule	Strongest type of attraction between polymer and dye
Wool	A protein chain with -NH_3^+ groups at the end of side chains when dyed in acid solution		<i>ionic</i>
<i>polyester</i>		Few polar groups on dye molecule	<i>permanent dipole-permanent dipole</i>
Cotton		Several -NH_2 groups. Linear molecule	<i>hydrogen bonding</i>

[2]

- 2 The use of a standard hydrogen electrode for measuring standard electrode potentials is often not practicable. The diagram below shows a calomel electrode. This is often used in preference to the standard hydrogen electrode and has a standard electrode potential, $E^\ominus$, of +0.27 V.



calomel electrode

- (a) The electrode is based on mercury metal, Hg, in contact with a saturated solution of Hg₂Cl₂.

- (i) Suggest **one** advantage and **one** disadvantage of using a calomel electrode over a standard hydrogen electrode.

Advantage ..portable, and no hydrogen gas is needed.....

Disadvantage ..mercury is toxic (poisonous)..... [1]

- (ii) Give the oxidation state of mercury in Hg₂Cl₂.

oxidation state = ...+1..... [1]

- (b) A 25.0 g sample of Hg₂Cl₂ is vaporised at 400 °C and a pressure of 101 kPa.
A student assumes that the formula of the gaseous mercury chloride molecules is Hg₂Cl₂.

Calculate the volume of gas, in dm³, that would be expected under these conditions.

$$M_r(\text{Hg}_2\text{Cl}_2) = 2(200.6) + 2(35.5) = 472.2$$

$$n = 25.0 / 472.2 = 0.0529 \text{ mol}$$

$$V = nRT / p = (0.0529 \times 8.314 \times 673) / 101\,000$$

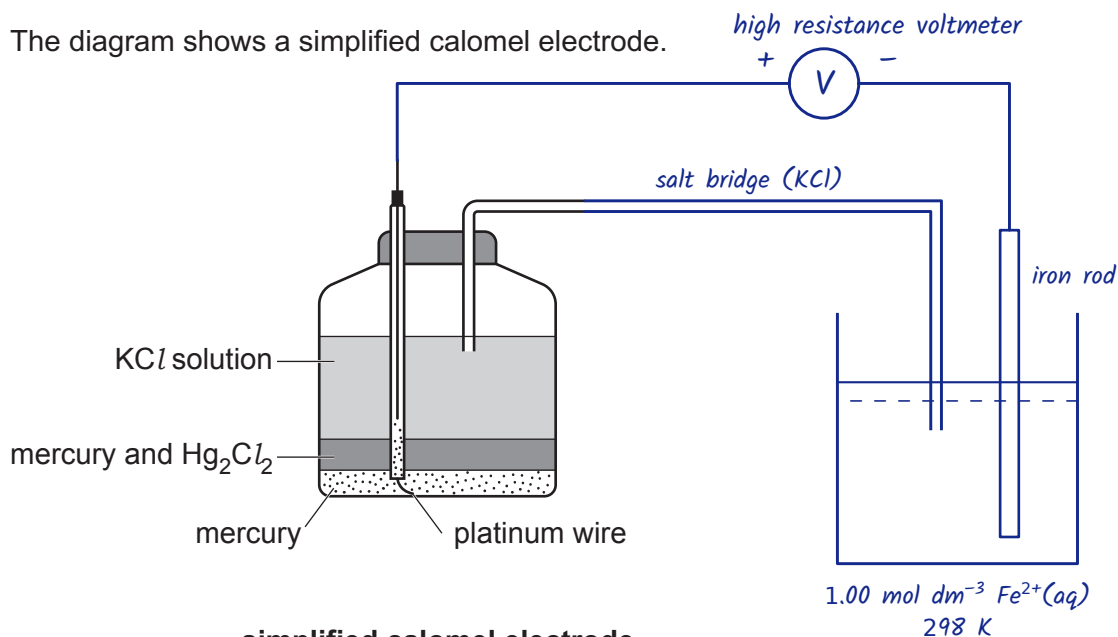
$$= 2.93 \times 10^{-3} \text{ m}^3 = 2.93 \text{ dm}^3$$

volume of gas = ...2.93..... dm³ [3]

Turn over

(c)* A student investigating the rusting of iron is given a calomel electrode.

The diagram shows a simplified calomel electrode.



The student wants to use this electrode to measure the standard electrode potential of a $\text{Fe}^{2+}(\text{aq})|\text{Fe}(\text{s})$ half-cell.

Give instructions on how to do this, justifying the uses of the pieces of apparatus you name. You may add to the diagram above to illustrate your answer.

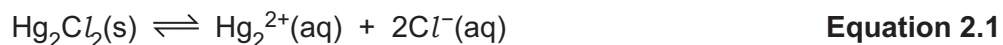
Use the calomel electrode as one half-cell. For the other half-cell put an iron rod (electrode) into $1.00 \text{ mol dm}^{-3} \text{ Fe}^{2+}(\text{aq})$, e.g. $\text{FeSO}_4(\text{aq})$, in a beaker, so that $\text{Fe}^{2+}(\text{aq}) + 2\text{e}^- \rightleftharpoons \text{Fe}(\text{s})$ can take place. Keep both at 298 K (25°C), since $E^\ominus$ changes with temperature and concentration.

Join the half-cells with a salt bridge (the side arm containing KCl solution, or filter paper soaked in $\text{KNO}_3(\text{aq})$) dipping into the Fe^{2+} solution; it completes the circuit and lets ions move to keep the charge in each beaker balanced without the solutions mixing.

Connect the platinum wire and the iron rod with leads to a high resistance voltmeter, so negligible current is taken and the ion concentrations stay the same.

Connect so the reading is positive: the calomel electrode is the positive electrode, joined to the + terminal, as iron is more negative. $E_{\text{cell}} = E(\text{positive}) - E(\text{negative})$, so $E^\ominus(\text{Fe}^{2+}|\text{Fe}) = +0.27 - E_{\text{cell}}$. E_{cell} is about 0.71 V , giving -0.44 V .

- (d) An equilibrium, represented by **equation 2.1**, exists between the solid Hg_2Cl_2 and its ions in solution.



The solubility of the solid Hg_2Cl_2 in a saturated solution at 298 K is $3.5 \times 10^{-4} \text{ g dm}^{-3}$.

Calculate the solubility product, K_{sp} , for Hg_2Cl_2 at 298 K. Include the units.

Give your answer to an **appropriate** number of significant figures.

$$M_r(\text{Hg}_2\text{Cl}_2) = 472.2$$

$$\text{solubility} = 3.5 \times 10^{-4} / 472.2 = 7.41 \times 10^{-7} \text{ mol dm}^{-3}$$

$$[\text{Hg}_2^{2+}] = 7.41 \times 10^{-7}; \quad [\text{Cl}^{-}] = 2 \times 7.41 \times 10^{-7} = 1.48 \times 10^{-6}$$

$$\begin{aligned} K_{\text{sp}} &= [\text{Hg}_2^{2+}][\text{Cl}^{-}]^2 = (7.41 \times 10^{-7}) \times (2 \times 7.41 \times 10^{-7})^2 \\ &= 1.6 \times 10^{-18} \text{ (2 s.f.)} \end{aligned}$$

$$\text{solubility product, } K_{\text{sp}} = \dots 1.6 \times 10^{-18} \dots \text{ units } \dots \text{mol}^3 \text{ dm}^{-9} \dots [5]$$

- 3 Iodine, I_2 , is an essential dietary element. The recommended maximum daily intake of iodine for an adult is $1.5 \times 10^{-4} \text{ g}$ ($150 \mu\text{g}$).

A group of chemistry students read that fish is a good source of iodine in the form of iodide ions. They decide to extract the iodine from 600 g of fish.

The students blend the fish in a food processor with 100 cm^3 of water, leave it to stand overnight and then filter the mixture into a beaker.

- (a) One of the students suggests that if they add silver nitrate solution they can confirm the presence of iodide ions in the solution.

- (i) Describe what the students would observe if the only halide ion present in the solution was the iodide.

a (pale) yellow precipitate forms [1]

- (ii) Write an **ionic** equation for this reaction. Include state symbols.



[1]

- (b) The students pour the filtered mixture into a separating funnel containing 20 cm^3 of hexane, 5 cm^3 of dilute sulfuric acid and 5 cm^3 of hydrogen peroxide solution.

Iodine is formed and dissolves in the hexane layer which goes purple. The purple layer is separated from the aqueous layer and transferred to a conical flask.

The purple coloured solution is titrated with standard $0.0010 \text{ mol dm}^{-3}$ sodium thiosulfate solution. The end point is indicated by the disappearance of the purple colour.

- (i) The hydrogen peroxide oxidises the iodide ions in the fish to iodine.

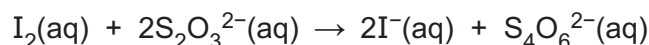
Write a half equation for this oxidation reaction.

Explain why this reaction is classified as oxidation.

Half equation *$2I^- \rightarrow I_2 + 2e^-$*

Explanation *iodide ions lose electrons (oxidation state of I rises from -1 to 0)*..... [1]

- (ii) The equation for the titration reaction is given below.



Name the element oxidised in this reaction. Give its oxidation state before and after the reaction.

Element oxidised *sulfur*.....

Oxidation state before reaction *+2*..... oxidation state after reaction *+2.5*..... [2]

- (iii) The students obtained an average titre of 5.30 cm^3 of $0.0010 \text{ mol dm}^{-3}$ sodium thiosulfate. Calculate the **mass** of iodine in μg in a **120 g** portion of fish.

Give your answer to **two** significant figures.

$$n(\text{S}_2\text{O}_3^{2-}) = 5.30 \times 10^{-3} \text{ dm}^3 \times 0.0010 = 5.30 \times 10^{-6} \text{ mol}$$

$$n(\text{I}_2) = 5.30 \times 10^{-6} / 2 = 2.65 \times 10^{-6} \text{ mol (in 600 g of fish)}$$

$$\text{mass of } \text{I}_2 = 2.65 \times 10^{-6} \times 253.8 = 6.73 \times 10^{-4} \text{ g}$$

$$\text{in 120 g: } 6.73 \times 10^{-4} / 5 = 1.345 \times 10^{-4} \text{ g} = 134.5 \mu\text{g}$$

$$= 130 \mu\text{g (2 s.f.)}$$

mass of iodine = ...130..... μg [4]

- (iv) One of the students suggests that the titre value is too small and will lead to an unacceptably high percentage error.

Calculate the percentage error based on the students' titre value.

$$\text{uncertainty} = 2 \times 0.05 = \pm 0.10 \text{ cm}^3; \quad 0.10 / 5.30 \times 100 = 1.9 \%$$

percentage error = ...1.9..... % [1]

- (v) Suggest how the experiment could be modified to improve the accuracy of the result.

.....Use a more dilute sodium thiosulfate solution (e.g. $0.00010 \text{ mol dm}^{-3}$) so.....
the titre is larger and its percentage error smaller (or extract the iodine.....
from a larger mass of fish)..... [1]

- (c)* The mass of the iodine in the hexane solution could also have been determined using colorimetry.

Suggest a suitable method that could be used to measure the mass of iodine in the hexane solution using a colorimeter or visible spectrophotometer.

Show how you would process the results.

Make up a range of at least 5 standard solutions of iodine in hexane of known concentration, by dissolving a weighed mass of iodine in hexane in a volumetric flask and diluting accurately.

Select the filter of the complementary colour to the purple solution, i.e. the one giving maximum absorbance (yellow-green, about 520 nm).

Zero the colorimeter with a cuvette of pure hexane (the solvent). Measure the absorbance of each standard, rinsing the cuvette with the next solution each time.

Plot a calibration curve of absorbance against concentration of iodine.

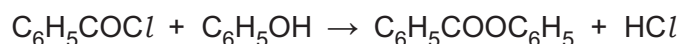
Measure the absorbance of the purple hexane layer from the fish with the same filter and cuvette, and read its concentration from the calibration curve.

Processing: moles of I_2 = concentration (mol dm^{-3}) $\times$ 20/1000 dm^3 of hexane (i.e. $\div 50$); mass of I_2 = moles $\times$ 253.8 g, then $\times 10^6$ to give μg ($\div 5$ for a 120 g portion).

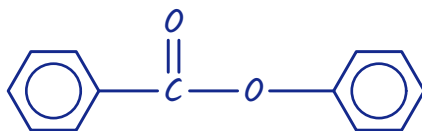
[6]

4 This question refers to the **Practical Insert** that is provided as an insert to this paper.

(a) The equation for the reaction producing phenyl benzoate is as follows:



(i) Draw a structural formula for phenyl benzoate, showing the bonding in the ester group.



[1]

(ii) Use the student results to calculate the percentage yield of phenyl benzoate obtained from the practical.

$$n(\text{phenol}) = (20.73 - 15.82) / 94.1 = 4.91 / 94.1 = 0.0522 \text{ mol}$$

$$1 : 1, \text{ so theoretical mass} = 0.0522 \times 198.2 = 10.34 \text{ g}$$

$$\text{actual mass} = 9.71 - 5.61 = 4.10 \text{ g}$$

$$\% \text{ yield} = 4.10 / 10.34 \times 100 = 39.65 \%$$

$$\text{percentage yield} = 39.65\% \quad [3]$$

(b) (i) In **step 8** of the procedure the water reacts with any remaining benzoyl chloride.

Write the equation for this reaction.



[1]

(ii) Suggest and explain the reason for **step 13** in the procedure.

..To test whether any unreacted phenol is left: neutral FeCl_3 turns purple.....
 ..with phenol..... [1]

- (iii) Describe the practical procedure used to measure the melting point of an organic solid. You **do not** need to discuss the type of melting point apparatus you use.

Seal one end of a melting point (capillary) tube in a Bunsen flame. Tap a small amount of the dry, powdered solid to the bottom of the tube and pack it down. Put the tube in the apparatus and heat so the temperature rises slowly near the melting point; record the temperature when the solid starts to melt and when it has completely melted. [3]

- (iv) What information can the students get from their melting point?

It shows the purity: it melts at 66 to 68 °C, lower than and over a range below 70 °C, so it contains impurities. [1]

- (v) The recrystallisation procedure uses ethanol as the solvent.

Give the key properties needed by a solvent to be effective in recrystallisation.

The solute (product) must be very soluble in the hot solvent but have low solubility in the cold solvent. [1]

- (c) The students carry out thin layer chromatography of the phenyl benzoate formed. One student states that this will enable them to assess the purity of their product.

Comment on the validity of this statement.

You should describe any possible observations to back up your comments.

The statement is only partly valid. If more than one spot shows on the TLC plate (under UV), the phenyl benzoate is impure, e.g. a spot at the same R_f as a phenol reference shows unreacted phenol; one spot suggests it may be pure. However TLC cannot show how pure or impure the product is: it is qualitative, not quantitative, so it cannot give the % by mass of phenyl benzoate.

[4]

END OF QUESTION PAPER

[illegible]

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