



Oxford Cambridge and RSA

# A Level Chemistry B (Salters)

H433/03 Practical skills in chemistry

Practical Insert

**Wednesday 20 June 2018 – Morning**

**Time allowed: 1 hour 30 minutes**



## INSTRUCTIONS

- Do not send this insert for marking; it should be retained in the centre or destroyed.

## INFORMATION

- This document consists of 4 pages. Any blank pages are indicated.

## Preparing an ester

An A level chemistry class is given a practical which requires them to prepare a sample of the ester, phenyl benzoate. They are also asked to calculate the percentage yield they achieve and to assess the purity of their product.

The students carry out the experiment as shown in the procedure below.

### Procedure

#### Preparation of phenyl benzoate

1. Transfer about 5.0 g of solid phenol into a weighing bottle and weigh it to the nearest 0.01 g.
2. Pour 90 cm<sup>3</sup> of 2 M sodium hydroxide into a conical flask and add the phenol from the weighing bottle.
3. Reweigh the weighing bottle to the nearest 0.01 g.
4. In a fume cupboard pour 9 cm<sup>3</sup> of benzoyl chloride into the conical flask.
5. Insert the bung securely and shake the bottle for 15 minutes, carefully releasing the pressure every few minutes as the flask gets warm.
6. Cool the flask under cold, running tap-water.
7. Filter the crude product using a suction filtration apparatus.  
Use a spatula to break up the lumps of ester on the filter paper, being careful not to puncture the filter paper.
8. Pour more water over the crude ester to remove any remaining benzoyl chloride.

#### Recrystallisation

9. Transfer the crystals to a boiling tube and just cover them with ethanol.
10. Place the boiling tube in a water-bath or beaker of hot water, kept at about 60 °C and stir with a glass rod.
11. If some solid ester is still visible, add just enough ethanol to dissolve it completely after stirring.
12. In order to allow the separation of the ester as a solid rather than an oily liquid (phenyl benzoate has a low melting point) add more ethanol to double the volume of solution.
13. Place one drop of the solution onto a white tile and add one drop of neutral iron(III) chloride solution.
14. Cool the solution in an ice-water mixture until crystals appear.
15. Filter the crystals through the suction apparatus using a clean Buchner funnel and filter paper. To avoid losing any solid break the vacuum and use the filtrate to rinse the boiling tube into the funnel.
16. Using suction again rinse the crystals with about 1 cm<sup>3</sup> of cold ethanol and drain thoroughly.
17. Press the crystals between sheets of filter paper to remove excess solvent. Then put the crystals on another dry piece of filter paper and place in a warm oven for an hour.
18. Weigh the dry crystals in a pre-weighed specimen bottle and record the mass of your sample of phenyl benzoate.
19. Using melting point apparatus determine the melting point of your crystalline sample.

*[Reference: Modified from 'Independent Learning Project for Advanced Chemistry; More functional groups – ILPAC unit 03', Inner London Education Authority, first published 1984 by John Murray (Publishers) Ltd]*

**Results**

Mass of weighing bottle and phenol/g	20.73
Mass of weighing bottle after emptying/g	15.82
Mass of specimen bottle/g	5.61
Mass of specimen bottle and phenyl benzoate/g	9.71
Melting point of product/°C	66–68

**Other observations**

Melting point of phenyl benzoate from data book = 70 °C

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**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										
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.

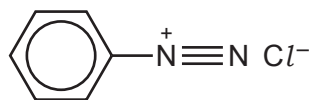
## INFORMATION

- 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.



**benzenediazonium chloride**

Name the reagents and conditions needed to make this compound.

Reagents

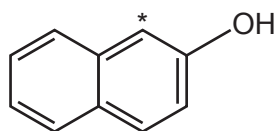
.....

Conditions

..... [3]

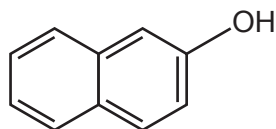
(b) Naphthalen-2-ol, shown below, is used to make the dye. A piece of cotton is dipped into naphthalen-2-ol dissolved in sodium hydroxide. The diazonium solution is then added to dye the cotton red.

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.

.....  
.....  
.....  
.....  
.....  
.....  
.....  
..... [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,  $\text{NO}_2$ , groups are attached.

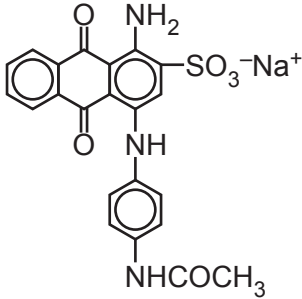
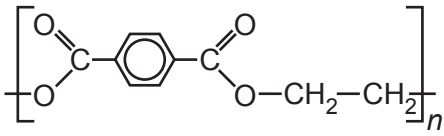
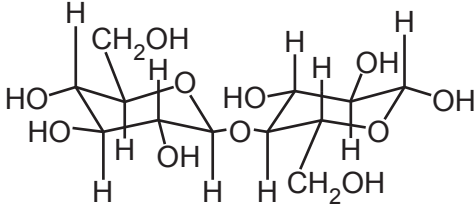
..... [1]

(ii) Give a **different** property affected by the attachment of sulfonate,  $\text{SO}_3^-$ , groups.

..... [1]

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

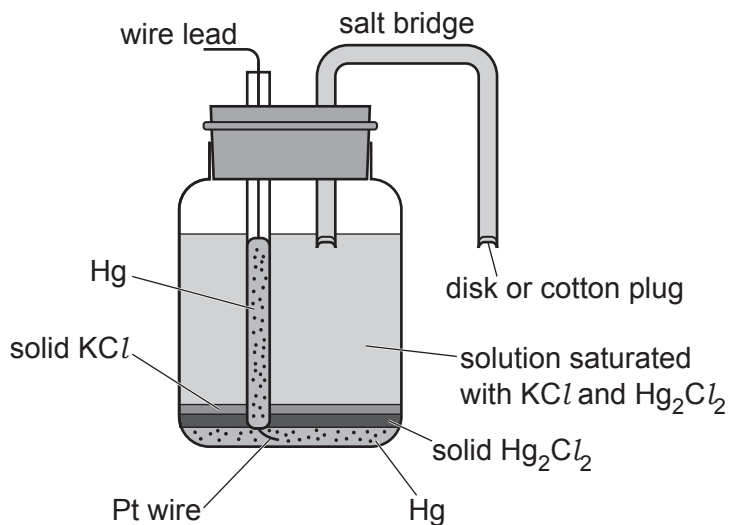
Use your knowledge of molecular interactions to fill in the empty boxes in the following table.

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 $-\text{NH}_3^+$ groups at the end of side chains when dyed in acid solution		
		Few polar groups on dye molecule	
Cotton		Several $-\text{NH}_2$ groups. Linear molecule	

[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 Hg<sub>2</sub>Cl<sub>2</sub>.

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

Advantage .....

Disadvantage ..... [1]

- (ii) Give the oxidation state of mercury in Hg<sub>2</sub>Cl<sub>2</sub>.

oxidation state = ..... [1]

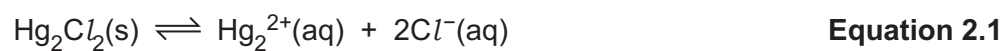
- (b) A 25.0 g sample of Hg<sub>2</sub>Cl<sub>2</sub> is vaporised at 400 °C and a pressure of 101 kPa. A student assumes that the formula of the gaseous mercury chloride molecules is Hg<sub>2</sub>Cl<sub>2</sub>.

Calculate the volume of gas, in dm<sup>3</sup>, that would be expected under these conditions.

volume of gas = ..... dm<sup>3</sup> [3]



- (d) An equilibrium, represented by **equation 2.1**, exists between the solid  $\text{Hg}_2\text{Cl}_2$  and its ions in solution.



The solubility of the solid  $\text{Hg}_2\text{Cl}_2$  in a saturated solution at 298 K is  $3.5 \times 10^{-4} \text{ g dm}^{-3}$ .

Calculate the solubility product,  $K_{\text{sp}}$ , for  $\text{Hg}_2\text{Cl}_2$  at 298 K. Include the units.

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

solubility product,  $K_{\text{sp}} = \dots\dots\dots$  units  $\dots\dots\dots$  [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  $100 \text{ cm}^3$  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.

..... [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  $20 \text{ cm}^3$  of hexane,  $5 \text{ cm}^3$  of dilute sulfuric acid and  $5 \text{ cm}^3$  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.

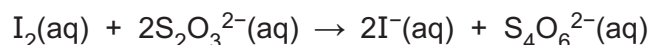
Write a half equation for this oxidation reaction.

Explain why this reaction is classified as oxidation.

Half equation .....

Explanation ..... [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 .....

Oxidation state before reaction ..... oxidation state after reaction ..... [2]

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

mass of iodine = .....  $\mu\text{g}$  [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.

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

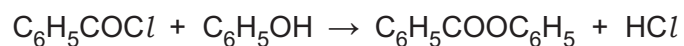
- (v) Suggest how the experiment could be modified to improve the accuracy of the result.

.....  
.....  
..... [1]



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.

percentage yield = ..... % [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.

.....  
 ..... [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.

.....  
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.....  
..... [3]

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

.....  
..... [1]

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

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

.....  
..... [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.

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..... [4]

**END OF QUESTION PAPER**



**ADDITIONAL ANSWER SPACE**

If additional space is required, you should use the following lined page(s). The question number(s) must be clearly shown in the margin(s).

A large area of lined paper for writing. It features a vertical solid line on the left side, creating a margin. The rest of the page is filled with horizontal dotted lines, providing space for writing answers.



Blank writing area with horizontal dotted lines and a vertical solid line on the left side.

A large vertical rectangular area on the left side of the page, bounded by a solid vertical line on the left and a dotted vertical line on the right. This area is filled with horizontal dotted lines, creating a series of approximately 20 rows for writing answers.



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