

**Extended Response Task 2 2024**

**Chromatography and Polarity - Validation**

**Total Marks: /50**

**Weighting 5%**

Name: \_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_\_

**Multiple Choice**: **(5 marks)**

1. Retention time can be used to identify a compound in a mixture using gas chromatography.

Which one of the following will not affect the retention time of a compound in a gas

chromatography column?

a) concentration of the compound  
b) nature of the stationary phase  
c) rate of flow of the carrier gas  
d) temperature of the column

2. The paper chromatograph of four dyes, G, P, R and Y, is shown below.

A diagram of a graph

Description automatically generated

The Rf value of the dye most strongly adsorbed onto the stationary phase is

a) 0.25

b) 0.33

c) 0.75

d) 0.78

3. A thin layer chromatography (TLC) plate was set up with hexane as the mobile phase and silica gel as the stationary phase. The chromatogram below was obtained.

A black dot and blue lines

Description automatically generated

Which one of these best describes the polarity of the components?

**Compound Stationary phase Mobile phase**

a) polar polar polar

b) non-polar polar non-polar

c) non-polar non-polar non-polar

d) non-polar non-polar polar

4. One use of thin layer chromatography (TLC) is in the analysis of urine samples taken from professional athletes. TLC can be used to detect whether any illegal substances are present in the urine. Consider the two TLC plates below which have been run under identical conditions. On the left is a control plate, showing the markers for the illegal substances cocaine, methamphetamine and heroin. On the right is a test urine sample.

*Control plate Test urine sample*

*solvent front*

4.8 cm

2.5 cm

*solvent front*

cocaine

methamphetamine

heroin

6.7 cm

5.1 cm

1.8 cm

3.5 cm

Has the urine sample tested positive for any of the illegal substances?

1. Yes, cocaine.
2. Yes, methamphetamine.
3. Yes, heroin.
4. No, none.

5. Thin layer chromatography (TLC) was performed on five (5) different essential oils; bergamot, eucalyptus, lavandula, orange and pine. The TLC plate is shown below, along with the corresponding Rf value for each resolved component.

*bergamot eucalyptus lavandula orange pine*

Rf = 0.883

Rf = 0.5

Rf = 0.5

Rf = 0.233

Rf = 0.367

Rf = 0.267

Rf = 0.267

Rf = 0.667

Rf = 0.567

Rf = 0.417

Rf = 0.667

A small amount of an unknown essential oil, labelled as ‘Sample X’, was then analysed by TLC using identical running conditions. This plate is shown below. Use the data provided to determine which **two** essential oils have been mixed to produce ‘Sample X’.

solvent front = 5.5 cm

4.86 cm

3.12 cm

2.75 cm

1.28 cm

1. Bergamot and pine.
2. Eucalyptus and lavandula.
3. Orange and eucalyptus.
4. Lavandula and pine.

**Short Answer (45 marks)**

6. The catalytic converter in a car takes in the undesirable gases produced by the combustion of petrol, such as carbon monoxide and various nitrogen oxides, and converts them into less harmful gases such as carbon dioxide, nitrogen gas and water. The less harmful gases are released into the atmosphere as we drive.

NOx catalytic N2

hydrocarbons converter H2O

CO CO2

Metals such as platinum, palladium and rhodium are used as the catalyst in a catalytic

converter.

a) The table below shows the major gaseous products to exit a catalytic converter.

Complete this table by drawing the electron dot diagram for each of the three gases,

representing all valence shell electron pairs either as : or –. In addition, state the shape of

the molecule, and indicate whether or not the molecule is a polar molecule. (9 marks)

|  |  |  |  |
| --- | --- | --- | --- |
|  | **Electron Dot Diagram** | **Molecular Shape** | **Polar molecule (‘yes’ or ‘no’)** |
| N2 |  | **linear** | **No** |
| CO2 |  | **linear** | **No** |
| H2O |  | **v-shaped / bent** | **Yes** |

A sample of exhaust fumes from a car was analysed by gas chromatography (GC) to determine the composition of the fumes and assess the effectiveness of the catalytic converter.

b) Briefly describe the principles of gas chromatography and state one reason why GC may have been chosen over other forms of chromatography to perform the analysis in this situation. (4 marks)

* **gas chromatography involves passing a sample of gas through a thin tube which is packed with an inert substance (such as silica) coated in a viscous non-volatile liquid**
* **an inert carrier gas such as helium acts as the mobile phase and moves the gas through the chromatography column where the various gas components move at different speeds as they interact with the stationary phase**
* **the gases separate based on their size/molar mass as well as their polarity and can be identified via their retention times**
* **GC works best for separating gaseous mixtures or mixtures that can easily be vaporised, which is why it is suitable for analysing car fumes**

The data below shows GC standards for four of the relevant gases, as well as the chromatogram obtained from analysis of the car exhaust fumes.

*The two chromatograms below, for methane and carbon monoxide, were obtained from the analysis of car exhaust fumes that* ***do not pass*** *through a catalytic converter.*

*The two chromatograms below, for nitrogen and carbon dioxide, were obtained from the analysis of exhaust fumes released from an* ***efficiently functioning*** *catalytic converter.*

*The chromatogram for the sample of exhaust fumes being analysed is shown below.*

c) Elaborate on the information provided by this data, in terms of the composition of the car exhaust fumes and the effectiveness of the catalytic converter. (3 marks)

* the car exhaust fumes contain the same nitrogen levels as an efficiently functioning catalytic converter.
* whilst all of the methane has been removed by the converter, a small amount of carbon monoxide is still present in the exhaust fumes, however this is greatly reduced from the levels seen without a catalytic converter.
* the amount of carbon dioxide is comparable to, although slightly lower than, an efficiently functioning converter, presumably due to the small amount of carbon monoxide still present in the exhaust fume mixture.

7. High performance liquid chromatography (HPLC) has many useful applications, with one of the most common being in the monitoring and analysis of additives used in food and beverages. A common additive found in many low calorie food and drink products is the artificial sweetener known as ‘aspartame’. The safety of aspartame has been studied extensively over the years, with many people reporting side effects such as nausea, dizziness and abdominal pain. Whilst it is still classified as a ‘safe substance’ by various food and health organisations, many people choose to avoid aspartame consumption.

A chemist decided to run some tests to compare the aspartame levels found in four different types of soft drink, and she used HPLC to perform the analysis.

a) Discuss the chemical principles behind the process of HPLC by answering the following questions.

1. What physical properties must a sample have to make it appropriate for HPLC analysis to be used? (2 marks)

* **samples can not be in gaseous form**
* **samples must be soluble to a degree in the particular solvent being used so that a liquid sample can be prepared**

1. Describe how HPLC is able to separate the components of a sample, in particular focussing on the role of polarity in the process. (4 marks)

* **either a polar solvent (mobile phase) is used with a non-polar stationary phase or a non-polar solvent (mobile phase) can be used with a polar stationary phase**
* **the sample to be analysed/separated is dissolved in the mobile phase and moves through the HPLC column**
* **the components of the sample will adhere to the stationary phase with different strengths due to the varying polarity of each component (also components will have varying degrees of interaction with the mobile phase)**
* **the components therefore move along the column at differing rates and exit the column at different times, where they can be collected/identified/further analysed**

The chemist used previous HPLC data to produce the ‘control’ chromatogram shown below, which displays the retention times for several common soft drink additives, including aspartame.

She then ran HPLC analysis on samples of four (4) different soft drinks; Pepsi, Diet Pepsi, Pepsi Max and Fanta Orange. The individual chromatograms for each soft drink are shown below. You may assume they were carried out under the same HPLC conditions as the ‘control’ above.

b) Which of the soft drinks contained aspartame? Name the drink with the highest aspartame concentration. (2 marks)

* Diet Pepsi, Pepsi Max, Fanta Orange contained aspartame
* Diet Pepsi contained the highest level

The chemist wanted to quantify her data, so she ran HPLC on a series of aspartame standards to produce a calibration curve. The results obtained from this are summarised in the table below.

|  |  |
| --- | --- |
| **Aspartame concentration**  **(g L-1)** | **Absorbance units** |
| 0.1 | 70 |
| 0.2 | 135 |
| 0.3 | 200 |
| 0.6 | 410 |

c) Plot the calibration curve for aspartame on the grid below. (5 marks)

**Calibration Curve for Aspartame**

* axis correctly labelled (1)
* x-axis (scale) (1)
* y-axis (scale) (1)
* data points (1)
* line of best fit (1)

d) List two (2) controlled variables the chemist would have had to consider when performing HPLC on the various aspartame standards. (2 marks)

any 2 of…

* same temperature
* same solvent
* same stationary phase
* same type of HPLC column
* same pressure used
* i.e. tests run under same conditions
* take absorbance readings at same wavelength

8. Below is the detector read-out from a high performance gas chromatography apparatus analysing the organic residues inside a chemical reaction tank using a polar stationary phase in the column. The mobile phase used was helium which had a column retention time of

0.6 minutes, as seen from the graph.

A graph showing different types of substances

Description automatically generated

a) Which compound in the tank was present in the greatest concentration? (1 mark)

Propane was present in the largest concentration as the peak is highest.

b) Which compound had a Retention Factor of 0.34? Show calculations. (3 marks)

* Ts = 1.75 minutes

- This time corresponds to the substance dimethyl ether.

c) Which compound being tested is the least polar? Explain. (3 marks)

* Methane is the least polar
* A low polarity molecule would not be attracted to the polar stationary phase and so would pass through the instrument more quickly

- Methane takes the shortest time, apart from the mobile phase (1.15 minutes), hence is least attracted

d) By considering the bonding types, explain why the Retention Time for ethanol would

be the greatest. (2 marks)

* Ethanol is highly polar as it can form hydrogen bonds
* This would make it highly attracted to the polar stationary phase and hence pass through the machine in the longest time.

9. Analysis of various hair products such as dyes, sprays and serums can be of great benefit to forensic investigators. Since hair samples are often found at crime scenes, identification of the hair products which are coating the hair can provide valuable information to investigators.

One common and effective method used for the analysis of hair products is thin layer chromatography (TLC). The TLC plate below shows the analysis of six (6) different popular brands of hair spray.

A diagram of hair loss

Description automatically generated

A hair sample from a crime scene was analysed by TLC and found to be coated in the hairspray ‘Style it up’.

a) Calculate the retention factor (Rf) values for each of the components (labelled W, X, Y and Z on the TLC plate) found in the hairspray ‘Style it up’. (4 marks)

|  |  |
| --- | --- |
|  | Rf |
| W | accept between 0.68 - 0.71 |
| X | accept between 0.51 - 0.53 |
| Y | accept between 0.42 - 0.45 |
| Z | accept between 0.20 - 0.22 |

Hair samples were then taken from three (3) suspects who had been arrested. The coatings on their hair samples were analysed by TLC. You may assume this analysis was performed under conditions identical to the original plate.

The results of the suspects’ TLC analyses are shown below.

**Miss White Mrs Peacock Miss Scarlet**

A row of measuring rulers

Description automatically generated with medium confidence

Based on the data provided by these TLC analyses;

b) Which suspect is most likely to have been at the scene of the crime? (1 mark)

Miss White

**End of Validation**