2411/305
INSTRUMENTAL METHODS OF
ANALYSIS
Oct./Nov. 2022
Time: 3 hours

THE KENYA NATIONAL EXAMINATIONS COUNCIL

DIPLOMA IN ANALYTICAL CHEMISTRY

INSTRUMENTAL METHODS OF ANALYSIS

3 hours

INSTRUCTIONS TO CANDIDATES

You should have the following for this examination:
  Answer booklet;
  Non-programmable scientific calculator.
This paper consists of TWO sections; A and B.
Answer ALL the questions in section A and any THREE questions from section B in the answer booklet provided.
Each question in section A carries 4 marks while each question in section B carries 20 marks.
Maximum marks for each part of a question are as shown.
*Candidates should answer the questions in English.*

This paper consists of 9 printed pages.

Candidates should check the question paper to ascertain that all
the pages are printed as indicated and that no questions are missing.

Turn over

SECTION A (40 marks)

Answer ALL the questions in this section.

1. The absorbance of a solution is 1.75 in the absence of stray radiation. Calculate the absorbance of the solution in the presence of 20% stray radiation. (4 marks)

2. Explain which of the molecules **A, B & C** below absorbs radiation of the longest wavelength. (4 marks)

A.

(B)

(C)
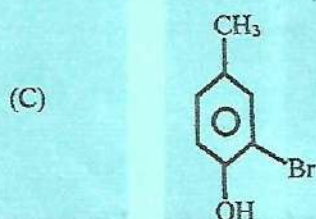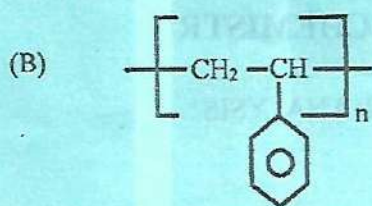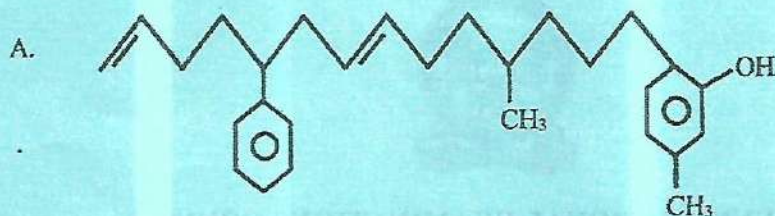
3. (a) An aqueous solution of potassium dichromate exists in equilibrium with chromate ions as $Cr_2O_7^{2-}{}_{(aq)} + H_2O \rightleftharpoons 2CrO_4^{2-}{}_{(aq)} + 2H^+{}_{(aq)}$. Explain the type of deviation from Beer's law exhibited by the aqueous solution of potassium dichromate. (2 marks)

   (b) Explain how the deviation from Beer's law can be minimized. (2 marks)

4. The $-O-H$ bond in an alcohol has a reduced mass of $4.70 \times 10^{-24} g/atom$ and that of $-O_1^2D \, bond$ is $8.90 \times 10^{-24} g/atom$. The absorption peak of the $-OH$ bond is $3200 \, cm^{-1}$. Calculate the position of the absorption peak of the $-O_1^2D$ bond. (4 marks)

5. The resolution curves in figures 1 and 2 were obtained when the same sample was analysed by a gas-liquid chromatograph using an open tubular column and a packed column. Explain which resolution peaks were produced by each column. (4 marks)
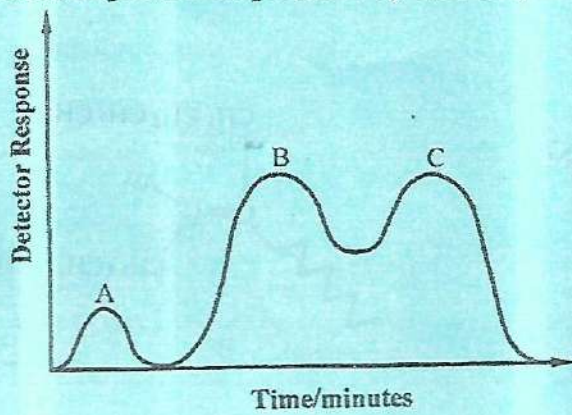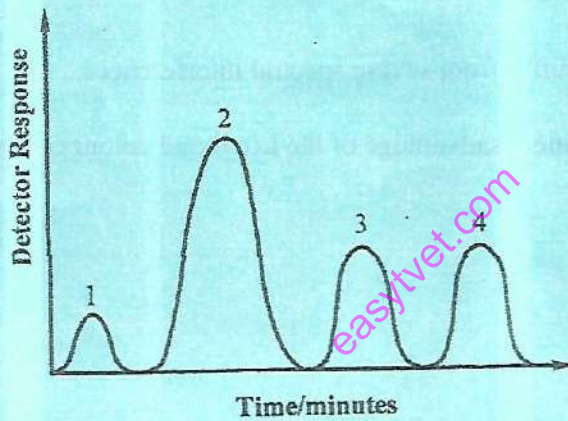

Fig. 1

Fig. 2

6. Figure 3 shows a sample preparation method used in gas chromatography. Describe how the sample is collected for analysis by GLC. (4 marks)
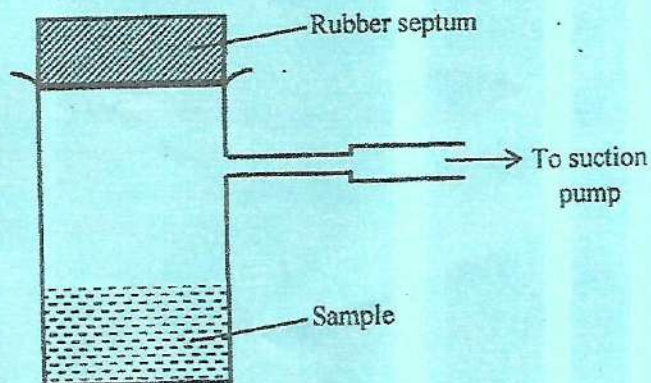

Fig. 3

7. Figure 4 shows the structures of alkenes A and B. The absorption peaks for the two molecules are at $1980\ cm^{-1}$ and $1845\ cm^{-1}$ respectively. Explain why the absorption peaks for the two alkene groups are not the same.   (4 marks)

Fig. 4

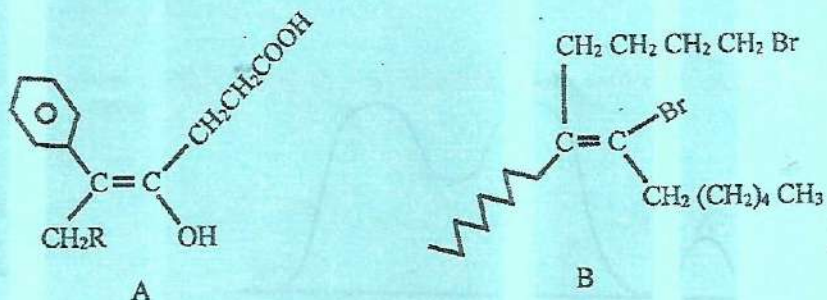8. Explain the observation that a salt of barium nitrate produces an intensively coloured radiation in the flame of a Bunsen burner while that of manganese (iii) nitrite does not.   (4 marks)

9. Explain why AAS does not suffer from severe spectral interferences.   (4 marks)

10. Explain **one** advantage and **one** disadvantage of the Lovibond colour comparator over photo-electric calorimeter.   (4 marks)

SECTION B (60 marks)

Answer any THREE questions from this section.

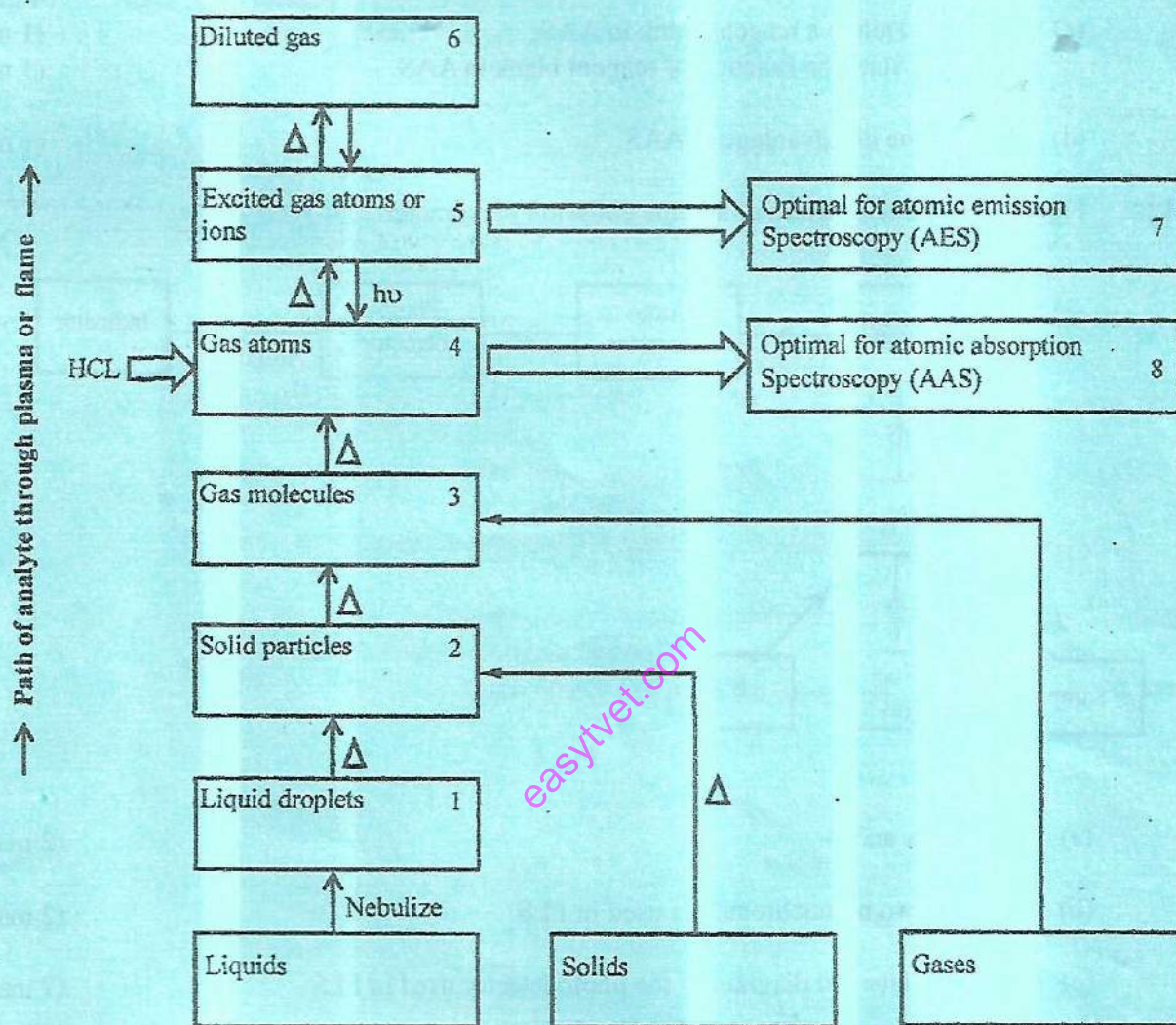11. Figure 5 shows the processes that take place in the burner of flame spectrometers.

Fig. 5

(a) (i) Indicate the form the analyte is likely to exist in boxes 1 to 6. (6 marks)

(ii) Write the equation that relates concentration and light power corresponding to emission (7) and absorption (8). (2 marks)

(iii) Write equations for the processes taking place in box:

(I) 1; (1 mark)

(II) 2; (1 mark)

(III) 3. (1 mark)

(iv) Write equations for the following processes:

      (I) absorption;   (1 mark)
      (II) emission;   (1 mark)

(b) List **four** methods of suppressing formation of oxides or molecular species in a flame.

                                                           (4 marks)

(c) (i) Define a reagent blank in AAS.   (1 mark)
      (ii) State the function of reagent blank in AAS.   (1 mark)

(d) State **one** disadvantage of AAS.   (1 mark)

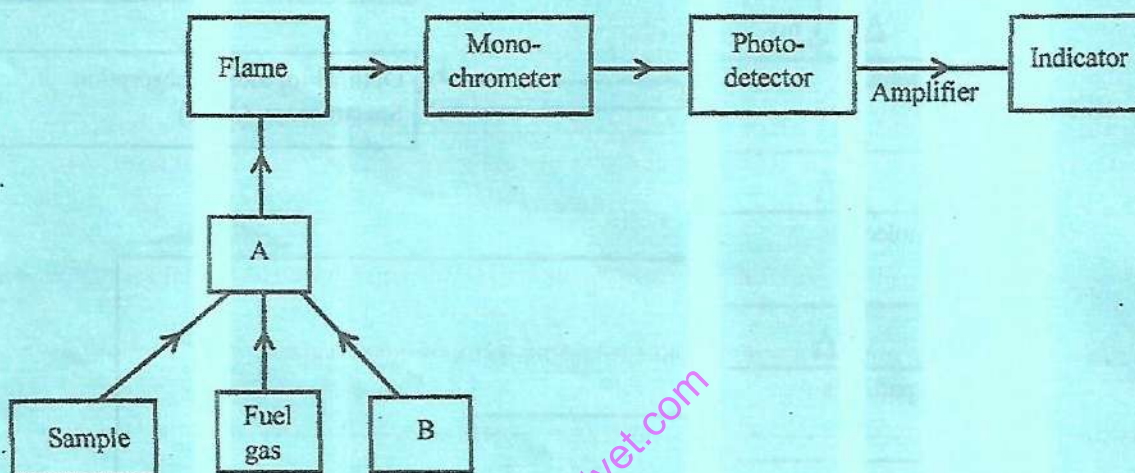12. Figure 6 is a bock diagram of a flame emission photometer.

Fig. 6

(a) Name **A** and **B**.   (2 marks)

(b) Name **two** monochromators used in FES.   (2 marks)

(c) Draw a labelled diagram of the photodetector used in FES.   (7 marks)

(d) (i) Name the part of the instrument labelled the indicator.   (1 mark)
      (ii) State the function of the part labelled the indicator.   (1 mark)

(e) State **three** function of a flame in flame spectroscopy.   (3 marks)

(f) The oxidant used in this instrument was nitrous oxide.

      (i) Name the type of flame being used.   (1 mark)
      (ii) Name **one** metal that is being investigated.   (1 mark)
      (iii) Name **two** other oxidants used in FES.   (2 marks)

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

(a) Indicate whether the following will affect the retention time of a compound in a GLC column:

(i) concentration of compound; (1 mark)
(ii) nature of stationary phase; (1 mark)
(iii) rate of flow of carrier gas; (1 mark)
(iv) temperature of column. (1 mark)

(b) Describe how retention time can be used to identify an unknown compound. (2 marks)

(c) Consider the following statements about a high performance liquid chromatography (HPLC) column that uses a polar solvent and a non-polar stationary phase. State whether the following statements is true or false. (4 marks)

Statement 1: polar molecules will be attracted to solvent particles.
Statement 2: non-polar molecules in solution will be attracted to stationary phase by dispersion forces.
Statement 3: polar molecules in solution will travel through the column more rapidly than non-polar moelecules.
Statement 4: Separation of non-polar molecules in stationary phase is by partition.

(d) A small sample of a mixture of esters passed through a GLC column attached to a mass spectrometer. The chromatogram showed two peaks A and B. The peak area and charge/mass ratio of the molecular ion of each compound are shown in Table 1:

Table 1

Methyl ester	Peak area	Mass to change ratio of molecular ion
A	1000	270
B	2000	298

(i) Explain the significance of the chromatogram having two peaks. (1 mark)
(ii) Calculate the relative amounts of each compound in the mixture. (4 marks)
(iii) State the molecules weights of compound A and B. (2 marks)

(e) Explain why hydrogen gas is used as a carrier gas in analysis of A and B: (3 marks)
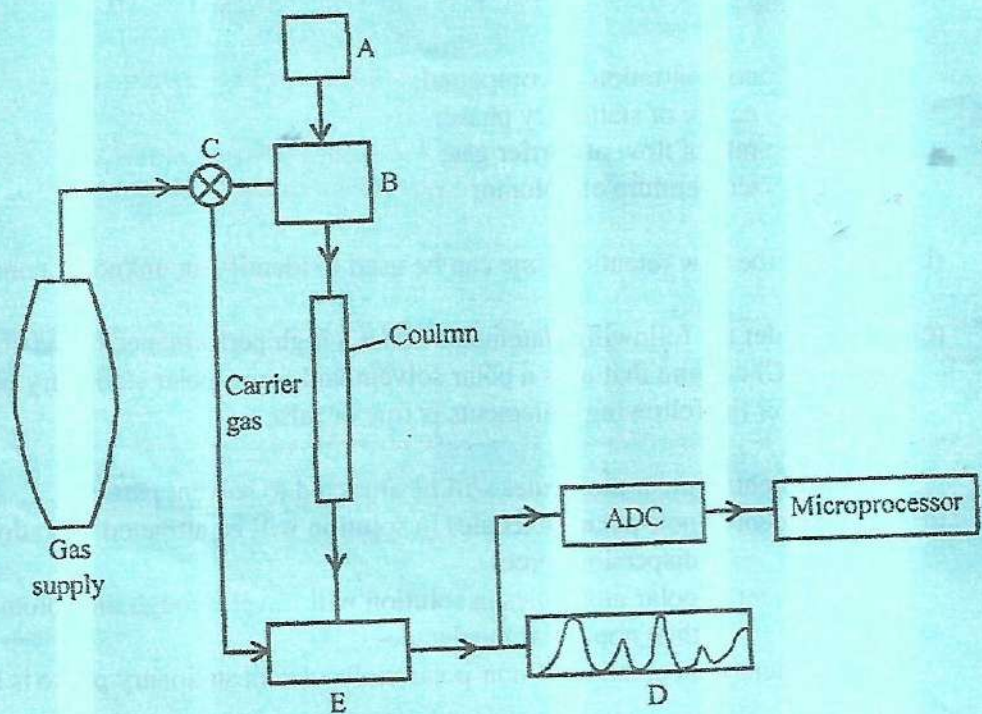
14. Figure 7 is a block diagram of a gas chromotograph.

Fig. 7

(a) Identify the components labelled A, B, C, D and E. (5 marks)

(b) Explain why the carrier gas is allowed to reach E before the sample. (1 mark)

(c) Explain each of the following features of the ovens used in gas chromatography:

    (i) maximum thermal gradient; (2 marks)
    (ii) proper insulation; (2 marks)
    (iii) low heating; (2 marks)
    (iv) low power consumption. (1 mark)

(d) Explain the following characteristics of a detector used in gas chromatography:

    (i) short response time; (2 marks)
    (ii) linear response to solutes; (2 marks)
    (iii) high sensitivity. (2 marks)

(e) State one advantage of the thermal conductivity detector. (1 mark)

15. (a) State Beer's law. (2 marks)

(b) Write the dilution formula using the standard symbols. (2 marks)

(c) 20 carbamazepine (formula weight = 236.26 g) tablets were found to weigh 10.000 g in total. The tablets were ground to a fine powder using a pestle and mortar and 0.300 g sample of the powder was boiled with 25 mL ethanol for a few minutes. The hot mixture was stirred in a closed flask for 10 minutes and filtered through sintered glass. The flask and filter were washed with ethanol and the washings combined with the filtrate. The cooled mixture was made upto 100 mL with ethanol. 5 mL of the ethanol extract was diluted to 250 mL with ethanol and absorbance of the resulting solution was found to be 0.588 absorbance units at a wavelength of 285 nm. Calculate the percentage of the active ingredient in a single tablet if the A1% 1 cm was 4900. (16 marks)

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