2411/305
INSTRUMENTAL METHODS
OF ANALYSIS
June/July 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;
a 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 indicated.
Candidates should answer the questions in English.

This paper consists of 7 printed pages.

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

SECTION A (40 marks)

Answer ALL the questions in this section.

1. A photometer with a linear response to radiation gave a potential reading of 685 mV with a blank in the light path and 179 mV when the blank was replaced by an absorbing solution. Calculate the absorbance of the solution. (4 marks)

2. A 500 cm^3 blood serum specimen was treated with reagents to generate colour with phosphate following which the sample was diluted to 1000 cm^3. Photometric measurement for the phosphate in a 25.00 cm^3 aliquot yielded an absorbance of 0.428. Addition of 1.00 cm^3 of a solution containing 0.0500 mg of phosphate to a second 25.00 cm^3 aliquot resulted in an absorbance of 0.517. Calculate the concentration of phosphate in mg/cm^3 of the original specimen. (4 marks)

3. (a) Explain why in the pressed pellet technique of preparing samples in IR spectroscopy, potassium bromide (KBr) is preferred to sodium chloride (NaCl) as a solvent. (3 marks)

   (b) Identify another solvent for the pressed pellet technique. (1 mark)

4. Figure 1 shows a burner used in AAS.

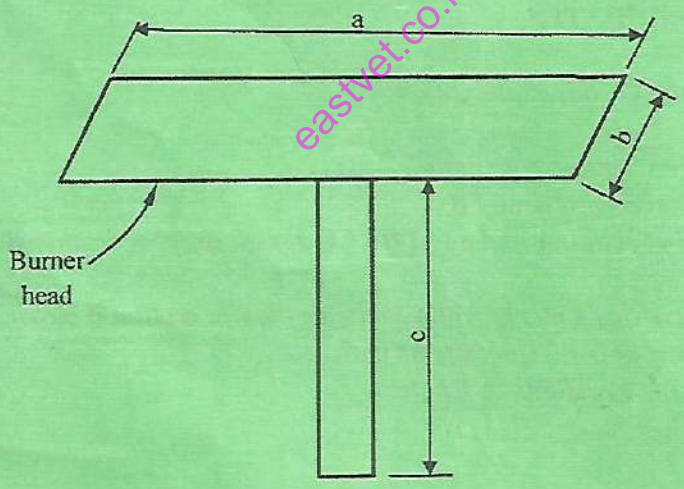


Fig. 1

   (a) Name the parts of the burner labelled **a** and **b**. (2 marks)

   (b) Explain the effect of rotating the burner head. (2 marks)

5. Figure 2 shows the vapour generation assembly for analysis of mercury by AAS.

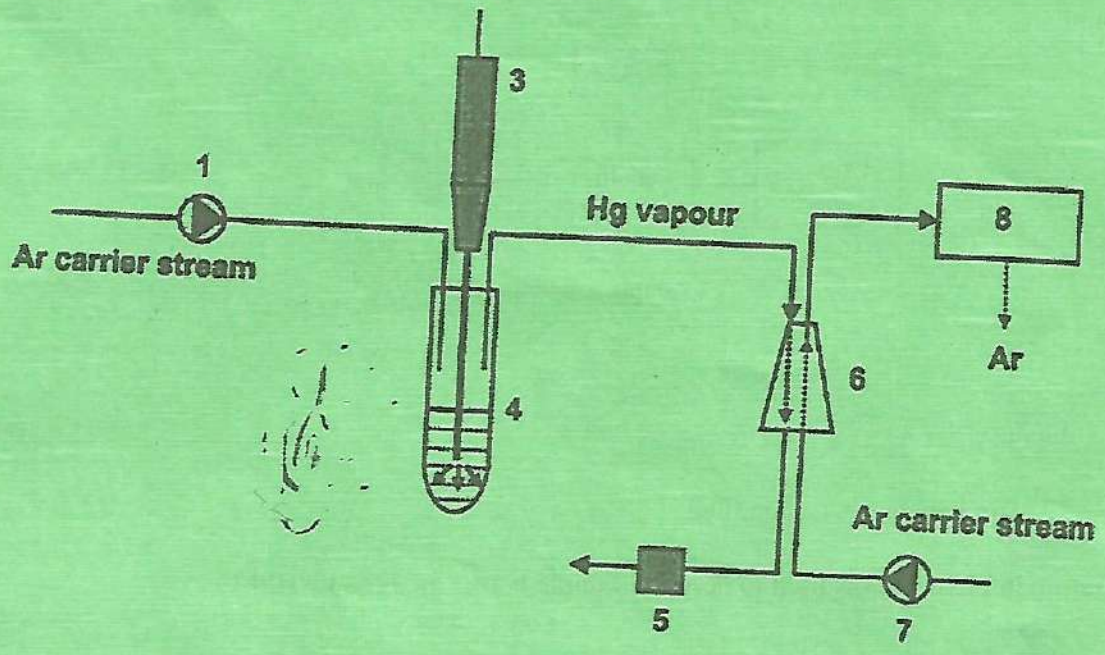


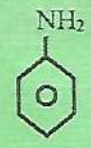
Figure 2

(a) State **two** functions of the argon (Ar) carrier gas in this set-up. (2 marks)

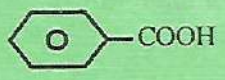
(b) Name the parts of the assembly labelled 3 and 6. (2 marks)

6. Compounds **A**, **B**, and **C** were separated in an HPLC column packed with silica gel. The mobile phase was 50/50 v/v mixture of ethylacetate and water. Explain the order in which the compounds were eluted. (4 marks)

A:



B:



C:

A =

$0.517 = 2 - \log T\%$

$-\log T\% = 2 - 0.517$

$-\log T\% = 1.483$

$-\log T = 30.41\%$

$T = 30.40\%$

$A = \varepsilon c l$

$0.517 = 30.41 \times c \times 1.0$

$\frac{0.517}{30.41} = \frac{30.41c}{30.41}$

$c = 0.017M$

7. The set-up in Figure 3 was used in a flame atomic spectroscopic estimation of magnesium in gypsum.

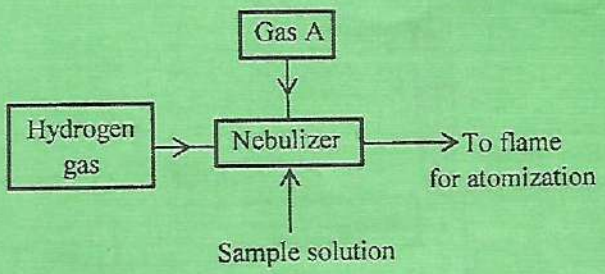


Fig. 3

(a) Identify gas **A**. (1 mark)

(b) Explain how gas **A** is identified. (3 marks)

8. The set-up in Figure 4 was used to de-gas a sample before analysis by HPLC.

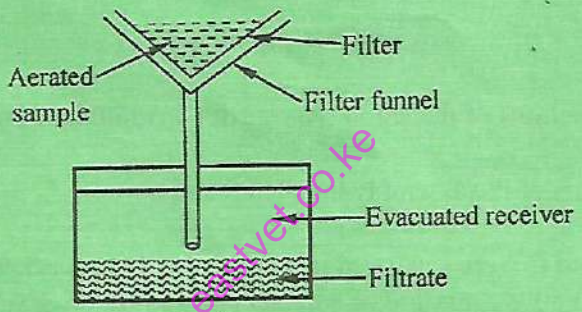


Fig. 4

(a) Explain why samples are de-gassed during analysis by HPLC. (2 marks)

(b) Explain how the de-gassing assembly works. (2 marks)

9. Identify the IR activity of each of the following molecules:

(a) SO_2Cl_2; (1 mark)
(b) ICl; (1 mark)
(c) O_3; (1 mark)
(d) H_2N-NH_2. (1 mark)

10. (a) State **two** ways of preparing solid samples for analysis by gas-liquid chromatography. (2 marks)

(b) State **two** properties of carrier gas used in GLC. (2 marks)

SECTION B (60 marks)

*Answer any **THREE** questions from this section.*

11. The Cu^{2+} ions form a bright blue complex with ammonia. The complex has maximum absorbance at a wavelength of 600 nm. A series of $Cu(NH_4)_4^{2+}$ solutions were prepared by adding various amounts of 0.025 M $CuSO_4$ to 2 cm^3 of ammonia. Distilled water was added to make the total volume of each solution to 7.00 cm^3. The percent transmittance of each solution was read using a blank which has only ammonia and water. The results are as shown in table 1.

Table 1

Sample	Volume of 0.025 M $CuSO_4$	T%	Molar concentration of Cu^{2+}	Absorbance
1	0.00 (blank)	100	0·0875	0.0000
2	1.00	66.1	0·01125	0·1798
3	2.00	41.7	0·02250	0·3799
4	4.00	18.6	0·05000	0.7305
5	5.00	12.3	0·06875	0.9101

(a) Calculate the molar concentration of Cu^{2+} in each solution and complete the table. (5 marks)

(b) Calculate the absorbance value of each solution and fill in the table. (5 marks)

(c) Use the results of (a) and (b) to plot a calibration graph. (7 marks)

(d) Use the calibration graph to determine the molarity of a Cu^{2+} solution which had a transmittance of 53.2%. (3 marks)

$A = 2 - \log T$

$2 - \log 53.2 = 0.2741$

From graph 0.013M.

12. (a) Metal complexes with low volatility are often difficult to analyse when performing atomic absorption measurements because the atomization efficiency is reduced to unacceptably low levels. Describe a method for eliminating complexing agents during analysis of non-volatile metal complexes. (5 marks)

(b) During analysis of metals by atomic absorption spectrophotometry, rubidium chloride is often added to both the standards and the samples. Explain the purpose of adding rubidium chloride to the sample solutions. (8 marks)

(c) (i) Explain 'matrix' as used in AAS. (2 marks)

(ii) Describe how 'matrix effect' is eliminated during estimation of metals by AAS. (5 marks)

13. (a) An IR spectrum reading is taken before and after treating acetone with the reducing agent, sodium borohydride, $NaBH_4$. Explain the peak readings that would be observed for the reactant acetone and for the product. (9 marks)

(b) After taking an IR spectrum of an organic compound synthesized in the laboratory, three major peaks were observed. Peak **A** has a 70% transmittance, peak **B** has a 50% transmittance and peak **C** has a 5% transmittance. The peaks have absorption peaks at 2500 cm^{-1}, 1700 cm^{-1} and 1200 cm^{-1} respectively. Sketch a labelled IR spectrum for this compound. (8 marks)

(c) Explain which of the peaks has higher absorbance. (3 marks)

14. Figure 5 is a diagram showing parts of a flame photometer.

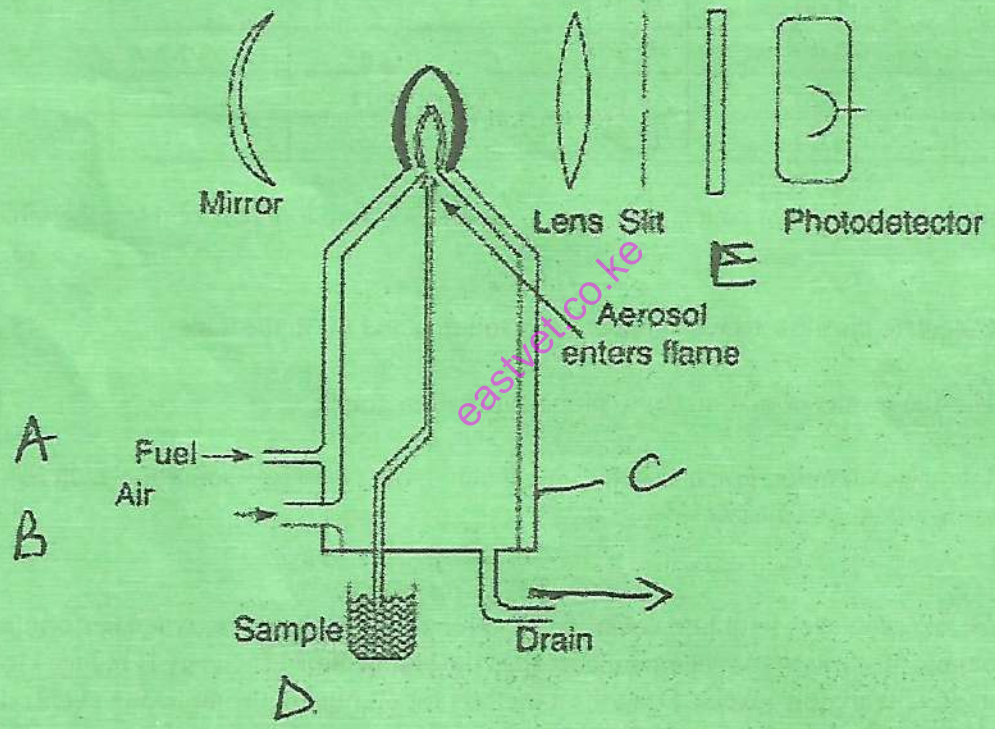


Figure 5

(a) Name the parts of the instrument labelled **A**, **B**, **C**, **D** and **E**. (5 marks)

(b) (i) Name the type of burner used in this flame photometer. (1 mark)

(ii) Give **two** reasons for the answer in (b)(i). (2 marks)

(iii) Explain **three** disadvantages of this type of burner. (6 marks)

(c) Name **three** major parts of a flame photometer not included in Figure 5. (3 marks)

(d) State the function of each of the following:

(i) mirror; (1 mark)
(ii) lens; (1 mark)
(iii) slit. (1 mark)

15. Figure 6 shows a type of detector used in gas chromatography.

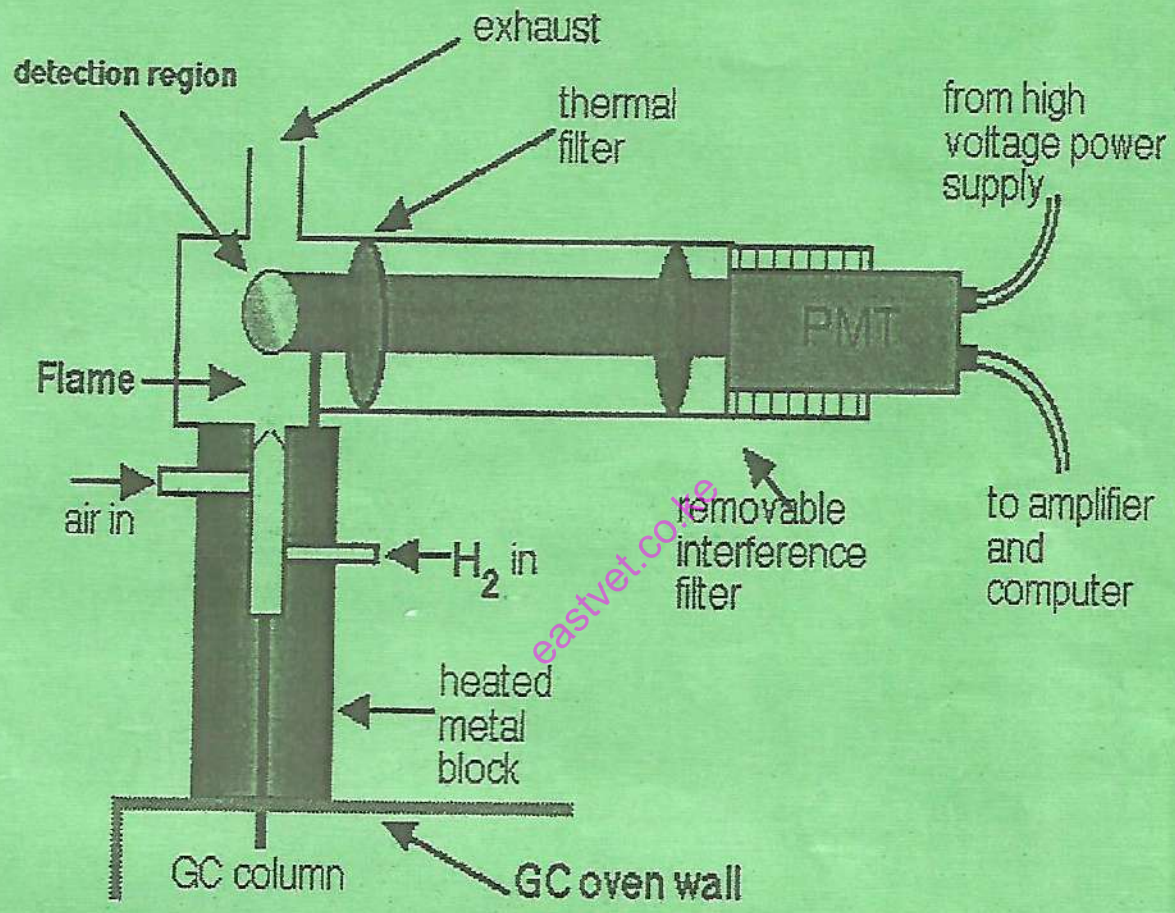


Figure 6

(a) (i) Write PMT in full. (1 mark)
(ii) Name the type of detector used in this instrument. (1 mark)
(iii) Describe how the analytical signal is produced in this detector. (7 marks)
(iv) Explain **four** advantages of this detector. (7 marks)

(b) Explain the function of the each of the following:

(i) GC oven; (2 marks)
(ii) thermal fitter. (2 marks)

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